

A simple synthesis of  $\gamma$ -aminopropionic acid. P. Šorm and  
J. Beránek (Časch. Akad. Věd. Prague, Czech.), Chem.  
Listy 47, 141-3 (1953).—A prepns. of  $H_2NCH_2CH_2CO_2H$   
(I) is described by the hydrogenation of  $NCCl_3$   
 $CO_2K$  (II) or  $NCCl_3CH_2CO_2K$  (III).  $ClCH_2CH_2CO_2K$   
(II) was obtained by refluxing I in EtOH 16 hrs.  
with KOH in 9% EtOH to II, m. 107° (from MeOH).  
Hydrogenation of 5 g. II in 25 ml. MeOH stnd. at 0° with  
Ni, over 14 g. Raney Ni at 0° and 110 atm. yielded,  
after 2 hrs., 2.8 g. (67%) I, m. 202°. The same product  
was obtained by hydrogenating 5 g. III in AcOH 25 ml.  
and 1 ml.  $H_2SO_4$  over 0.6 g. PtO<sub>2</sub> at 20° and 100 atm. for  
1 hr. and hydrolyzing the product with Ba(OH)<sub>2</sub>. Yield  
70%. M. Hudlický

*Re gear*

SORM, F.

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Steroids. Part 6.  $\beta$ -dimethoxyamino derivatives of steroids [in Russian with summary in English]. Sbor.Chekh.khim.rab. 18 no.6:842-853 D '53. (MLRA 7:6)

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Czechoslovak Academy of Science, Prague. (Steroids)

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HEROUT, V.; SORM, F.

Components of wormwood (*Artemisia absinthium L.*) and the isolation  
of a crystalline pro-chamazulogenogen [in English with summary in  
Russian]. *Sbor.Chekh.khim.rab.* 18 no.6:854-869 D '53. (MIRA 7:6)

1. Department of Natural Substances, Institute of Organic Chemistry,  
Czechoslovak Academy of Science, Prague. (Wormwood) (Chamazulogen)

SORM, F.; POKORNY, J.

Terpenes. Part 47. Synthesis of 1,7-dimethylazulene [abstract; in English].  
Sbor.Chekh.khim.rab. 18 no.6:885 D '53. (MLRA 7:6)

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Advances of chemistry of sesquiterpenes. Uspokhi Khim. 22, 564-82 '53.  
(CA 47 no.19:9944 '53) (MLRA 6:4)

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(MLRA 6:8)

1. Chekhoslovatskaya Akademiya nauk.  
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SO: Eastern European Accessions List, Vol. 3, No 11, Nov. 1954, L.C.

SORM, FRANTISEK

**Terpenes. XLVII. Synthesis of 1,7-dimethylazulene.**

Frantisek Sorm and Josef Pokorný (Prague, Czech.)  
*Chem. Listy*, 47, 153-9 (1953); *cl. C.A.* 47, 87016.--An unequivocal synthesis of 1,7-dimethylazulene was carried out by the following series of reactions: *Ei* 3-methyl-2-oxocyclopentanecarboxylate (I)  $\rightarrow$  *1/2* 3-methyl-2-oxo-1-(*t*-cyanopropyl)cyclopentanecarboxylate (II)  $\rightarrow$  *Ei* 3-methyl-2-oxocyclopentanecarboxylate (III)  $\rightarrow$  *Ei* 3-methyl-2-carbethoxymethylenecyclopentanecarboxylate (IV)  $\rightarrow$  *Ei* 3-methyl-2-carbethoxymethylenecyclopentanecarboxylate (V)  $\rightarrow$  the free acid (Va)  $\rightarrow$  8-methyl-*hept-1-ylo[3.3.0]decan-5-one* (VI)  $\rightarrow$  5,8-dimethylbicyclo[5.3.0]decan-1-ol (VII)  $\rightarrow$  5,8-dimethylbicyclo[5.3.0]decan-1-ene (VIII) which give 5,8-dimethylbicyclo[5.3.0]decan (IX) on hydrogenation and 1,7-dimethylazulene (X) on dehydrogenation. I (41 g.) (0.24 mole) was added in 3 portions to a suspension of 0.2 g. (0.27 mole) Na dust in 250 ml. PhMe, the mixt. refluxed 4 hrs. at 130°, the hot Na salt gel treated with 43.2 g. (0.27 mole) BrCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CN, heated 20 hrs. at 130°, decompd. with H<sub>2</sub>O, and distd. *in vacuo*, yielding 43.6 g. (77.6%) II, *b.p.* 128-30°. II (11.95 g.) hydrolyzed by refluxing with 200 ml. concd. HCl 10 hrs. yielded 17.24 g. (52%) 3-methyl-2-oxocyclopentanecarboxylic acid, *m.p.* 45-50° [semicarbazone, *m.p.* 151-5° (from EtOH)], which gave 75.5% III, *b.p.* 98-9°, by esterification in the presence of anhyd. HCl, and 91% III by azeotropic esterification [semicarbazone, *m.p.* 150° (from EtOH)]. III (39.4 g.) and 21 g. Zn wool in 80 ml. dried PhMe treated with 10 ml. BrCH<sub>2</sub>COEt, heated, treated with an addnl. 20 ml. BrCH<sub>2</sub>COEt, the mixt. refluxed 18 hrs., decompd. with ice

and 80 ml. 10% H<sub>2</sub>SO<sub>4</sub>, the org. layer extd. with ether, and the ext. washed with aq. NaHCO<sub>3</sub>, dried, and distd., yielded 22.5 g. (43%) IV, *b.p.* 155-60°. Hydrogenation of IV in AcOH over PtO<sub>2</sub> gave 95% V, *b.p.* 130-14°. Hydrolysis of 12.7 g. V with 120 ml. 10% NaOH under Na dust, gave 9.5 g. (95%) Va. The acid Va (2.28 g.) distd. with 2.3 g. Fe dust and 150 mg. powd. Ba(OH)<sub>2</sub> and the dil. filtrate washed with aq. K<sub>2</sub>CO<sub>3</sub> and again distd. gave 0.815 g. (34%) VI, *b.p.* 117-20° [semicarbazone, *m.p.* 236° (from MeOH)]. A Grignard reagent from 735 mg. Mg and 3.3. MeI in 23:1 ether treated with 1 g. VI in 8 ml. ether, the mixt. refluxed 5 hrs., decompd. with ice and NH<sub>4</sub>Cl, the distd. VII (200 mg.) dehydrated by heating 20 min. at 180° with 0.06 mg. KHSO<sub>4</sub>, and the mixt. chromatographed gave 178 mg. VIII, *b.p.* 97-100°. VIII (350 mg.) hydrogenated over 110 mg. PtO<sub>2</sub> in EtOH and AcOH gave 100 mg. IX, *b.p.* 76°. Dehydrogenation of 3.17 g. VIII with 1.87 g. S by heating 40 min. at 230° gave, after chromatography, 70 mg. blue X [C<sub>11</sub>H<sub>10</sub>(NO<sub>2</sub>) complex, *m.p.* 158°]. Infrared and ultraviolet spectra of X are given. XLVIII. Constitution of *α*- and *β*-cadinene. Vlastimil Herout and František Santavy. *Ibid.* 70-5.-By ozonization, excluding from ylang-ylang oil was identified as *5-isopropyl-2,8-dimethylbicyclo[4.1.0]decano* (I). On the basis of the reaction with MeMgBr and MeLi, *β*-cadinene is reported to be *5-isopropyl-3,8-dimethylbicyclo[4.1.0]deca-1,7-diene* (II). I in AcOEt ozonized 5.5 hrs. with 3% O<sub>3</sub> gave, after chromatography, approx. 45% of a diketone, C<sub>11</sub>H<sub>10</sub>, *m.p.* 102-3° [from C<sub>6</sub>H<sub>6</sub>-petr. ether (1:4)], believed to be *5-isopropylbicyclo[4.1.0]deca-2,9-dione* (III) [dioxime, *m.p.* 194-200° (from EtOH-petr. ether); disemicarbazone, *m.p.* 201 (H<sub>2</sub>O)]

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B(OH)).  $\delta$ -Cadinene isolated from citronella oil treated with peroxyphthalic acid in ether gave after evapn. of the ether and chromatography  $\delta$ -cadinene dioxide, m. 83.5-4.6° (from petr. ether),  $[\alpha]_D^{25}$  13.3°, and an oxide diol,  $C_{15}H_{20}O_2$ , m. 128.5-9.5° (from C<sub>6</sub>H<sub>6</sub>-petr. ether (3:1)),  $[\alpha]_D^{25}$  1.7°. The dioxide of II was refluxed with MeMgBr in ether 18 hrs., 120 hrs., and in dioxane 6 hrs.; in all 3 instances, unchanged starting material was recovered; refluxing the dioxide of II 48 hrs. with MeLi gave the same result. The infrared spectrum of III is given. XLIX. Sesquiterpenes of the cadinane type in Javanese citronella oil. Vlastimil Herout, Tibor Kolos, and Josef Pilva, *Ibid.* 440-8. Two pure sesquiterpenes,  $\gamma$ -cadinene and  $\delta$ -cadinene, have been isolated from *Javanese citronella oil* (I). The formula suggested for  $\gamma$ -cadinene in the literature (C.A. 27, 280) is challenged. No acyclic sesquicitronellene was found in the oil investigated. Fractionation and chromatography of I gave fractions contg.  $\delta$ -cadinene (II),  $d_{40}^{20}$  0.91-5,  $n_D^{20}$  1.5088,  $[\alpha]_D^{25}$  93.8°,  $\log \epsilon$  (246 mp) 2.65, hydrogenation of which gave cadinane,  $d_{40}^{20}$  0.8830,  $n_D^{20}$  1.4792,  $[\alpha]_D^{25}$  4.8°. II and HCl at -20° gave (-)-cadinene-2HCl, m. 117-18° (from B(OH)),  $[\alpha]_D^{25}$  -87.8°. Fraction contg.  $n_D^{20}$  1.4773,  $\gamma$ -Cadinene,  $d_{40}^{20}$  0.9125,  $n_D^{20}$  1.5078,  $[\alpha]_D^{25}$  147.0°,  $\log \epsilon$  (246 mp) 2.25. The cadinane and cadinene-2HCl prep'd. from  $\gamma$ -cadinene were identical with the products prep'd. from  $\delta$ -cadinene. M. Hudlický

SORM F.

Proteins. XIX. Methylation of chymotrypsinogen and chymotrypsin. p.245  
(Chemical Listy. Vol. 47, No 2, Feb. 1953) Czechoslovakia

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August 1953, Incl.

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"Reaction of Ketene with Acetals of Aldehydes and Ethyl Orthoformate," p 413,  
(Chemicke Listy, Vol.47, No.3, Mar. 1953, Praha.)

SO: Monthly List of East European Accessions, Vol.2, No.9, Library of Congress, September  
1953, Unclassified.

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" Steroids, Part 6. Steroid 3-Dimethyla-Mino-Derivatives," p. 418.  
(Chemicke Listy, Vol.47, No.3, Mar. 1953, Praha.)

SO: Monthly List of East European Accessions, Vol.2, No.9, Library of Congress, September  
1953, Uncl.

## CZECH

Amino acids and peptides. VIII. Peptides of 2,4-diaminobutyric acid. Milan Záoral, Josef Rudíkner, and František Šorm (Czech. Akad. Věd, Prague, Czech.). *Chem. Listy* 47, 427-30 (1953); cf. C.A. 48, 3003e. [In this abstr., R = carbobenzoyloxy throughout.] Several peptides and intermediates of L-H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH(NH<sub>2</sub>)CO<sub>2</sub>H (I) were prep'd. L-Glutamic acid gave by the Schmidt degradation, m. 215°; L-di-HCl salt, m. 196-6°; dipicrate, m. 181°; 2-(*tert*-butylsulfonate), C<sub>11</sub>H<sub>18</sub>N<sub>2</sub>O<sub>8</sub>S<sub>2</sub>Bu<sub>4</sub>I (the formula in the abstr. is wrong), m. 215° (from H<sub>2</sub>O). L-RNHCH<sub>2</sub>CH<sub>2</sub>CH(NH<sub>2</sub>)CO<sub>2</sub>H (II), m. 90-1°; hydrazide, m. 160-2°, prep'd. through II-Et ester or by treating 1-carbobenzoyloxy-3-carbobenzoylamino-2-pyrrolidinone (III) with 80% NaOH in H<sub>2</sub>O, soln. in EtOH. L<sub>2</sub>HCl (10 g.) in 240 ml. N NaOH with 12 g. CICO<sub>2</sub>CH<sub>2</sub>Ph (III) gave 7.5 g. (24.8%) II, m. 90-2°, 3.1 g. (30.6%) RNHCH<sub>2</sub>CH<sub>2</sub>CH(NH<sub>2</sub>)CO<sub>2</sub>H (IV), m. 234° (from H<sub>2</sub>O), and 3.7 g. (23.1%) I. Similarly, 3 g. H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH(NH<sub>2</sub>)CO<sub>2</sub>H.HCl in 47 ml. N NaOH and 2.8 g. II in 32 ml. N NaOH yielded 1.6 g. (31.5%) L-RNH(CH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>CO<sub>2</sub>H, m. 255° (from H<sub>2</sub>O). HCl salt of IV-Et<sub>2</sub>O (65.5% from IV with 8% HCl in EtOH), m. 152-3°. I (4 g.) was transformed into 3.3 g. (86.5%) IIIA, m. 143-144° (from AcOEt-petr. ether), which in turn was converted to 57.3% amide of II, m. 167° (from aq. AcOH). IV with COCl<sub>2</sub> in dioxane gave L-L-(2-carbobenzoylaminoethyl)-2,5-oxazolidinedione. The hydrazide of II (1.8 g.) in 20 ml. AcOH treated with 10 ml. HCl (1:10), 50 ml. Et<sub>2</sub>O, and, with cooling and stirring, 380 mg. NaNO<sub>2</sub> in 3 ml. H<sub>2</sub>O, the mixture poured into 50 ml. ice water, the ether layer washed with H<sub>2</sub>O, to remove the mineral acid, the dried ether soln.

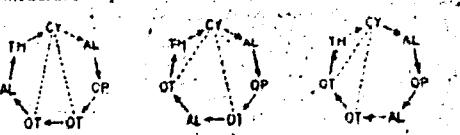
added dropwise to H<sub>2</sub>NCH<sub>2</sub>CO<sub>2</sub>Et in CHCl<sub>3</sub>, the mixt. let stand overnight, the solvents distd. off at 40° *in vacuo*, the residue dissolved in AcOEt, the soln. washed 3 times with HCl (1:5), 3 times with a satd. soln. of Na<sub>2</sub>CO<sub>3</sub>, and finally twice with 10% Na<sub>2</sub>CO<sub>3</sub> soln., dried, evapd. *in vacuo*, and the residue crystallized from aq. EtOH gave 1.6 g. (70.8%) L-RNHCH<sub>2</sub>CH<sub>2</sub>CH(NH<sub>2</sub>)CONHCH<sub>2</sub>CO<sub>2</sub>Et (V), m. 130-1°. also obtained (75%) by heating 0.4 g. II-A with 0.5 ml. H<sub>2</sub>NCH<sub>2</sub>CO<sub>2</sub>Et 5 min. at 135°. V is hydrolyzed with 0.5 NaOH to 79.5% free acid, m. 142-3°. V (600 mg.) in 5 ml. AcOH heated at 60° with a soln. prep'd. by mixing 3.1 g. 50% HI with 3.2 g. Ac<sub>2</sub>O, the PbCl<sub>2</sub> removed by Et<sub>2</sub>O extn., the iodine ppd. from the aq. layer as PbI<sub>2</sub>, the Pb ions removed with H<sub>2</sub>S, and the soln. evapd. several times to dryness yielded 194 mg. (98.2%) L-H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH(NH<sub>2</sub>)CONHCH<sub>2</sub>CO<sub>2</sub>H; dipicrolonate monohydrate, m. 202-3° (from aq. EtOH). II (1.8 g.) and L-BuCH(NH<sub>2</sub>)CO<sub>2</sub>Et (from 1 g. of the HCl salt) gave by the azide method 1.95 g. (82%) L-RNHCH<sub>2</sub>CH<sub>2</sub>CH(NH<sub>2</sub>)CONHCHBuCO<sub>2</sub>Et-L (VII), also prep'd. by the anhydride method by treating at -5° 2 g. II in 20 ml. CHCl<sub>3</sub>, 0.9 g. III, and 0.5 g. MeNC<sub>2</sub>H<sub>5</sub> with a CHCl<sub>3</sub> soln. of L-BuCH(NH<sub>2</sub>)CO<sub>2</sub>Et (from 1 g. HCl salt), letting the mixt. stand 14 hrs., and evapg. it *in vacuo* to give 1.05 g. (40%) VII, m. 108°; the free acid, m. 155° (from aq. EtOH), treated with III, gave 88% L-H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH(NH<sub>2</sub>)CONHCHBuCO<sub>2</sub>H [dipicrolonate, m. 225-7° (from H<sub>2</sub>O)]. L-RNHCH<sub>2</sub>CH<sub>2</sub>CH(NH<sub>2</sub>)CONHCH(CO<sub>2</sub>Et)CH<sub>2</sub>CO<sub>2</sub>Et-L, m. 130-1° (from aq. EtOH), prep'd. in 70.1% yield by the anhydride method from 4 g. II, 1.75 g. di-Et<sub>2</sub>glutamate-HCl, 1.8 g. III, and 1 g. MeNC<sub>2</sub>H<sub>5</sub>, gave by

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71. dicarbobenzoyloxydipeptide, m. 180-90° (from aq. EtOH), from which was obtained 91.2% L-*L*-diamino-*D,L*-glutamic acid monohydrate, m. 192-3° (from aq. EtOH). L-RNH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH(NHR)CH<sub>2</sub>CH<sub>2</sub>R-NHR-L)CO<sub>2</sub>Et, m. 140-2° (from aq. EtOH), prep'd. in 01.8 g. yield by the azide method and in 50.2% yield by the anhydride method, was hydrolyzed quantitatively to the free acid, m. 160-2° (from aq. EtOH), which, hydrogenated in 13.41 over 15% Pd-C, yielded 76% L-H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH<sub>2</sub>NH<sub>2</sub>)CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub> (triglycine dihydrate, m. 222° (from H<sub>2</sub>O); *taurine*, m. 228-7° (from H<sub>2</sub>O). The ester, m. L-RNH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH<sub>2</sub>NH<sub>2</sub>-L)CO<sub>2</sub>Et (1.7 g., prep'd. by the anhydride method) was hydrolyzed to the free acid, m. 142-3° (from aq. EtOH), which, hydrogenated in AcOH over Pd-C, gave 91% L-H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH<sub>2</sub>NH<sub>2</sub>)CO<sub>2</sub>H. *Urethane*, m. 213-15° (decompn.) (from H<sub>2</sub>O). L-RNH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH(NHR)Bu-L)-CO<sub>2</sub>Et, m. 166-9° (from aq. EtOH), prep'd. in 63% yield by the azide method and in 33% yield by the azide synthesis, L-*L*-dipropyl, to 74% free acid, m. 143-5° (from aq. EtOH), which with HI gave 87.5% L-H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH<sub>2</sub>NH<sub>2</sub>-L)CO<sub>2</sub>H(bis-(2-naphthalenylsulfonate), m. 190-1° (from H<sub>2</sub>O)). L-RNH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH(NHR)-CH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>Et-L)CO<sub>2</sub>Et, m. 163° (from aq. EtOH), prep'd. in 25.5% yield, was saponified to 84.6% free acid, m. 177-8° (from aq. EtOH), and decarbobenzoyloylated to L-H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH<sub>2</sub>NH<sub>2</sub>)CH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>H, decompn., m. 192-5°. It was transformed with NH<sub>3</sub>·H<sub>2</sub>O to 77% L-RNH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH(NH<sub>2</sub>)CONHCH<sub>2</sub>CONHNH<sub>2</sub>, m. 178-9° (from aq. EtOH).

aq. AcOH), which by the azide synthesis yielded 60.5% L-RNH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH<sub>2</sub>NHCOCH(NHR)CH<sub>2</sub>CH<sub>2</sub>R-NHR-L)CO<sub>2</sub>Et, m. 151-3° (from AcOEt-petr. ether). This was transformed to 92% corresponding hydrate, m. 202-3° (from aq. AcOH), which with H<sub>2</sub>NCH<sub>2</sub>CO<sub>2</sub>CH<sub>2</sub>Ph·HCl gave 44.5% L-RNH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH<sub>2</sub>NHCOCH(NHR)CH<sub>2</sub>CH<sub>2</sub>R-NHR-L)CONHCH<sub>2</sub>CO<sub>2</sub>CH<sub>2</sub>Ph, m. 203-5° (from aq. AcOH). Decarbobenzoylation by hydrogenation in AcOH on Pd-C liberated 78% L-H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH<sub>2</sub>NHCOCH(NHR)CH<sub>2</sub>CH<sub>2</sub>R-NHR-L)CONHCH<sub>2</sub>CO<sub>2</sub>CH<sub>2</sub>Ph, m. 215-17° (from H<sub>2</sub>O). IV (4 g.) in 10 ml. CHCl<sub>3</sub> treated at -65° with H<sub>2</sub>NCH<sub>2</sub>CO<sub>2</sub>CH<sub>2</sub>Ph (from 3.2 g. of its HCl salt), and 1.6 g. MeNC<sub>2</sub>H<sub>5</sub> in 5 ml. CHCl<sub>3</sub>, and finally with  $\text{P}_2\text{O}_{10}$ -(CO)<sub>2</sub>NCH<sub>2</sub>COCl (from 3.2 g. CH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>NCH<sub>2</sub>COCl) in 15 ml. CHCl<sub>3</sub> gave L-RNH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH(NHCOCH<sub>2</sub>NHCOCH<sub>2</sub>NHCOCH(NHR)CH<sub>2</sub>CH<sub>2</sub>R-NHR-L)CONHCH<sub>2</sub>CO<sub>2</sub>CH<sub>2</sub>Ph, m. 230-1° (from aq. AcOH). *Ester*, prep'd. similarly in 44.5% yield, m. 188-9° (from aq. AcOH). IX. Constitution of phalloidin.

2. Bedřich Meloun, Bořivoj Keil, and František Šerný, Czech. Akad. Věd, Prague, Czech., *Chem. Listy* 47, 1504-10 (1953); cf. C.A. 47, 3237b.—New expts. with phalloidin show the sequence of L-threonyl-L-cysteinyl-L-alanyl-L-dihydroxyprolyl in the mol. The following possibilities for the structure of phalloidin are suggested:



OT = hydroxytryptophanyl, TH = threonyl, CY = cysteinyl, AL = alanyl, OP = dihydroxyprolyl. The dotted lines show the possible S bridges. m-hydroxytryptophan, m. 242°, R<sub>1</sub>:H<sub>2</sub>O 0.70, BuOH-AcOH-H<sub>2</sub>O (4:1:5) 0.34, PhOH contg. 25% H<sub>2</sub>O 0.86, BuOH-BuOH-H<sub>2</sub>O (4:1:5) 0.48. M. Hudlický

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SO: Monthly List of East European Accessions, LC., Vol. 3, No. 1, Jan. 1954, Unclassified.

SORM, F.

**Synthesis of methylenecycloalkanes.** Erantilok Sorm and M. Hudlický (Czech. Akad. Věd, Praha, Czech.). *Chem. Listy* 47, 708-11 (1953).—Methylenecycloalkanes were prep'd. by the decarboxylation of the corresponding cycloalkenylacetic acids. The acids contg. 5-, 6-, and 7-membered rings were obtained by the condensation of the cyclic ketones with  $\text{NCCH}_2\text{CO}_2\text{H}$  (I), the acids with 8- and 9-membered rings by the Reformatskii synthesis with  $\text{Br}-\text{CH}_2\text{CO}_2\text{Et}$  (II). *Methylenecyclopentane*, b. 72°,  $d_{4}^{20}$  0.7852,  $n_D^{20}$  1.4314. A mixt. of 3.2 g. cyclohexylidene- and 1-cyclohexenylacetic acids (cf. *C.A.* 34, 3076) gently heated at approx. 100° in an app. connected to a Dry Ice trap, and the crude product distd. with Na gave 1.8 g. (81%) methylenecyclohexane, b. 98°,  $d_{4}^{20}$  0.8043,  $n_D^{20}$  1.4512. *Cycloheptanone* (5 g.), 12 g. I, and 12 g.  $\text{C}_6\text{H}_5\text{N}$  heated 2 hrs. at 100-5°, and the mixt. extd. with  $\text{Et}_2\text{O}$ , the ext. washed with 10%  $\text{H}_2\text{SO}_4$ , 10% aq.  $\text{Na}_2\text{CO}_3$ , and  $\text{H}_2\text{O}$ , dried, and distd. in  $\text{H}_2\text{O}$  gave 2.7 g. (45%) *cycloheptylideneacetonitrile*, b. 105-12°, which, refluxed 27 hrs. with 2.4 g. KOH in 20 ml.  $\text{H}_2\text{O}$ , gave a mixt. of 1.9 g. (61.5%) *cycloheptylidene-* and *1-cycloheptenylacetic acids*, b. 143-7°, b. 149°. Decar-

boxylation of this mixt. yielded 70% *methylene cycloheptane*, b. 133°,  $d_{4}^{20}$  0.8257,  $n_D^{20}$  1.4585. *Cyclooctanone* (1 g.), 4 g. II, 1.57 g. Zn shavings, 10 ml.  $\text{C}_6\text{H}_6$ , and 8 ml. PhMe heated to 109° to start the reaction, then refluxed 2 hrs. at 105°, cooled, shaken with 10%  $\text{H}_2\text{SO}_4$ , the org. layer sep'd., stripped of the solvents, and the residue (4 g.) treated 2 hrs. with dry HCl at 103°, gave, after distn., 2.15 g. (45%) *Et cyclooctylidenacetate* (III), b. 132-7°, b. 135°. Hydrolysis of 2 g. III with 0.25 g. Na in 25 ml. 90%  $\text{EtOH}$  gave, after refluxing 4 hrs., a mixt. of 1.35 g. (78%) *cyclooctylidene-* and *1-cyclooctenylacetic acids*, b. 165°. Decarboxylation of this mixt. gave 61% *methylene cyclooctane*, b. 163°,  $d_{4}^{20}$  0.8485,  $n_D^{20}$  1.4083. *Cyclononanone* (6.8 g.), 8.1 g. II, and 3.2 g. Zn heated in 19 ml.  $\text{C}_6\text{H}_6$  and 18 ml. PhMe 2 hrs. at 110-20° and 8 hrs. at 80-90°, gave 2.5 g. (24.5%) *Et cyclononylideneacetate*, b. 131-3° (88%), gave 62% *methylene cyclononane*, b. 169°,  $d_{4}^{20}$  0.8652,  $n_D^{20}$  1.4808.

M. Hudlický

FAJKOS, J., SORM, F.

"On Steroids. VII Synthesis of 3-Hydroxyandrosta 5, 16-Diene-16-Carboxylic Acid"  
p. 712, (CHEMICKÉ LISTY, Vol. 47, no. 5, May 1953, Praha, Czechoslovakia).

SO: Monthly List of East European Accessions, LC, Vol. 2, No. 11, Nov. 1953, Uncl.

*SOPM, Frantisek*

Temeneš, L. Contribution to the constitution of elemol. Václavín Štoka, Vlastimil Herout, Josef Pliva, and František Sorm. (Czech. Akad. Věd, Praha, Czech.). *Chem. Listy* 47, 889-98 (1953); *Collection Czechoslov. Chem. Commun.*, 19, 124-34 (1954) (in English); cf. C.A. 47, 8704h. Comparison of the infrared spectra of elemol and of synthetic 1,1-dimethyl-2-sec-butyl-4-isopropylcyclohexane (I) contradicts the Ruzicka-van Veen formula (C.A. 24, 607) of elemol (II). Dehydrogenation of tetrahydroelemene (III) to 1-methyl-2,4-diisopropylbenzene (IV) proves that I has the skeleton of 1-methyl-1-ethyl-2,4-diisopropylcyclohexane (V). II isolated from the distn. residues of citronella oil by vacuum distn. was chromatographed and purified through its phenylurethan, m. 111-12°, to give pure II, m. 52.5-3.5°. Dehydrogenation of 40 g. tetrahydro-II (obtained by hydrogenation of II over PtO<sub>2</sub>) by heating with 220 g. 85% HCO<sub>2</sub>H 1 hr. on the steam bath gave, after chromatography and distn., 31 g. III, b.p. 128-30°. Heating 2.1 g. III and 0.95 g. S 7 hrs. at 180°-240° gave 0.95 g. IV, b.p. 100-5°, d<sub>4</sub> 0.8563, n<sub>D</sub> 1.4045. Quant. ozonization of II indicated 1.89 double bonds. I was synthesized as follows: refluxing

21 g. 2,2-dimethylcyclohexanone in 210 ml. CC<sub>4</sub> with 37.2 g. N-bromosuccinimide under ultraviolet illumination 40 min. gave 37 g. 2,2-dimethyl-6-bromocyclohexanone, m. 55.5-7° (from petr. ether); the dehydrobromination of which (3.5 g.) with 250 ml. colliding gave 11.9 g. 2,2-dimethyl-5-cyclohexenone (VI), b. 173-82° (decompn.). Refluxing 1 hr. 11.9 g. VI with a soln. obtained from 10.7 g. Mg and 31.4 g. iso-PrCl in H<sub>2</sub>O gave, by way of the semi-carbazone, m. 157-8°, 2,2-dimethyl-3-isopropylcyclohexanone, (VII), b.p. 103-5.5°, d<sub>4</sub> 0.8002, n<sub>D</sub> 1.4340. VII (1.03 g.) in petr. ether refluxed 1 hr. with a soln. of sec-BuLi made from 1.5 g. Li and 12 ml. sec-BuCl, gave, after chromatography, 0.6 g. 1-sec-butyl-2,3-dimethyl-5-isopropylcyclohexanone, b.p. 98-9°, the dehydration of which with HCO<sub>2</sub>H yielded 1,1-dimethyl-2-sec-butyl-4-isopropylcyclohexene (VIII), b.p. 105-8°. Hydrogenation of VIII in AcOH over PtO<sub>2</sub> gave I, d<sub>4</sub> 0.8412, n<sub>D</sub> 1.4001. Dehydrogenation of VIII with S (7 hrs. at 180°-210°) gave a compd. distg. at 27

mm. at bath-temp. (200°) was synthesized as follows: propylbenzene (IX) was synthesized from 2.4 g. Li and 14 g. (n<sub>D</sub> 1.4990)(2.3 g.) and sec-BuLi from 2.4 g. Li and 14 g. sec-BuCl refluxed 1 hr. in petr. ether gave 2.5 g. crude 1-sec-butyl-2-methyl-5-isopropenyl-2-cyclohexanol, b.p. 94-13°, which was aromatized by boiling with HCO<sub>2</sub>H to 0.25 g. 1-methyl-2-sec-butyl-4-isopropylbenzene (X), b.p. 98-101°, d<sub>4</sub> 0.8614, n<sub>D</sub> 1.4904. The same compd. was obtained as follows: carvomenthone (3 g.) refluxed 75 min. with sec-BuLi (from 1.5 g. Li and 12 ml. sec-BuCl) in petr. ether yielded, after chromatography, 1-sec-butyl-2-methyl-5-isopropylcyclohexanol, b.p. 87-91°. This heated with 90% HCO<sub>2</sub>H gave 1-sec-butyl-2-methyl-5-isopropylcyclohexene (I), b.p. 102-15°, which was dehydrogenated with S at 180-210° to 0.03 g. X, b.p. 103-8°, d<sub>4</sub> 0.8027, n<sub>D</sub> 1.4028. 1-Methyl-2,4-diisopropylbenzene (XI) was synthesized as follows: carvomenthone (d<sub>4</sub> 0.9124, n<sub>D</sub> 1.4764)(3.1 g.), refluxed 90 min. with iso-PrLi, prep'd. from 2.8 g. Li and 19 ml. iso-PrCl, gave 3.5 g. 1-methyl-2,4-diisopropylcyclohexanol, b.p. 70-81°. This (2 g.) was dehydrated with HCO<sub>2</sub>H to 1.34 g. of the compd., b.p. 88-90.5° which yielded, by heating 0 hrs. with S at 180-240°, 0.7 g. XI, b.p. 104-8°, d<sub>4</sub> 0.8073, n<sub>D</sub> 1.4946. LI. The composition of the chamazulene. Preliminary communication, P. Šorm, J. Novák, and V. Herout. *Chem. Listy* 47, 1097-9 (1953); *Collection Czechoslov. Chem. Commun.* 18, 527-9 (1953) (in English).—On the basis of all known data on the elementary analyses of chamazulene and its derivs., on the basis of oxidation products, and of infrared spectra detns., the correct formula for chamazulene, 1,4-dimethyl-7-ethoxydienone, C<sub>11</sub>H<sub>16</sub>, is suggested. LI. The structure of laserpitine. František Šorm, Miroslav Holub, and Vlastimil Herout. *Chem. Listy* 47, 1400-1403 (1953); *Collection Czechoslov. Chem. Commun.* 19, 133-40 (1954) (in German).—Laserpitine (I), the bitter principle of the root of *Laserpitium latifolium*, is a diester of angelic acid and laetard (II) which seems to be a bicyclic tetracydroxy ketone. Out of the 6 O atoms, 3 are bound to three neighboring C atoms, and 2 to two adjacent C atoms in some other part of the mol. I. isolated by petr. ether extn., m. 117°, [α]<sub>D</sub> 119°, mol. wt. (Rast) 446, hydrogenated over PtO<sub>2</sub> in AcOH gave tetrahydro-laserpitine, m. 83-5° (from petr. ether). Sapon. of 2.8 g. I with 6.2 ml. 1.2 N Ba(OAc)<sub>2</sub> in 10 ml. MeOH at room temp. gave,

*di-Me norcaryophyllenate*,  $b_4$  100-2°, 70 g.; *di-Me caryophyl-*  
*lenate*,  $b_4$  120-1.5°, and 40 g.; *di-Me homocaryophyllenate*,  
 $b_4$  130-2°,  $n_D^{20}$  1.4403,  $[\alpha]_D^{20}$  32.08. Refluxing 37.4 g. II 4  
 hrs. with 10 ml. MeOH and a few drops of  $H_2SO_4$  gave 12.8 g.  
 (30.0%) *Me II homocaryophyllenate*,  $b_4$  186° (III). III  
 (3.2 g.) was treated with  $SOCl_2$  in 30 ml.  $C_6H_6$ , the solvent  
 evapd. *in vacuo*, the ester acid chloride add. with  $C_6H_6$  and  
 added to a benzene soln. of 1.25 equiv.  $Me_2Cd$  to give, after  
 heating 10 min., decompr., with 10%  $H_2SO_4$ , and ether  
 extn., 2.60 g. (81%) *Me*  $\beta$ -[2,2-dimethyl-1-acetyl(cyclobutyl)-  
 propionate (IV),  $b_4$  132-8°; *semicarbazone* of the free keto  
 acid, m. 105-6°. To 14 g.  $HgCl_2$ -activated Zn in 70 ml.  
 $C_6H_6$  were added 6.5 g. IV and 14 g.  $BrCH_2CO_2Me$  in 30 ml.  
 $C_6H_6$ , the mixt. boiled 10 min., decompr. with 10%  $H_2SO_4$ ,  
 and extd. with  $Et_2O$ , the residue dehydrated by boiling 2  
 hrs. with 40 ml.  $Ac_2O$  to give, after chromatography, 2.43  
 g. *Me*  $\beta$ -[2-(2-carbomethoxyethyl)-3,3-dimethylcyclobutyl]croto-  
 nate,  $b_{44}$  110-17°, which hydrogenated over  $PtO_2$  in  $AcOH$  gave  
*Me*  $\beta$ -[2-(2-carbomethoxyethyl)-3,3-dimethylcyclo-  
 butyl]butyrate, 1.18 g.,  $b_4$  156-84°;  $\beta$ -[2-(2-carbomethoxy-  
 ethyl)-3,3-dimethylcyclobutyl]butyric acid,  $b_4$  190° (bath  
 temp.) (obtained by alk. sapon. of the ester) (0.65 g.) distd.  
 with 0.65 g. Fe dust, and 0.15 g.  $Ba(OH)_2$  gave 260 mg.  
 (54%) 2,8,8-trimethylbicyclo[0.2.5]nonan-4-one (V),  $b_4$  130°  
 (bath temp.); *semicarbazone*, m. 176-7°. Another prepn.  
 started with IV (3.53 g.) which was refluxed 5 hrs. with 1.88  
 g.  $NCC_6H_5CO_2Et$ , 1.2 g.  $AcOH$ , 1.2 g.  $AcONa$  and 5 ml.  
 $C_6H_6$ , to give 2.31 g. (45%) *Et*  $\alpha$ -cyano- $\beta$ -[2-(2-carbomethoxy-  
 ethyl)-3,3-dimethylcyclobutyl]crotonate,  $b_4$  134-6°, which  
 hydrogenated over Pd on  $CaCO_3$  yielded 2.15 g. *Et*  $\alpha$ -cyano- $\beta$ -  
 [2-(2-carbomethoxyethyl)-3,3-dimethylcyclobutyl]butyrate,  
 $b_{44}$  160-3° (VI). Heating 1.98 g. VI 15 hrs. with 10 ml.  
 $HCl$  and 5 ml.  $AcOH$  yielded 1.83 g. (98%)  $\beta$ -[2-(2-carbo-  
 oxyethyl)-3,3-dimethylcyclobutyl]butyric acid,  $b_{44}$  180°, which  
 yielded by cyclization 48% V,  $b_4$  112-14°. The *semicar-  
 basone* of V (160 g.), 460 ml. Na and 0 ml.  $Bu_4N^+$  were heated  
 20 hrs. at 200°, the mixt. dilut. with  $H_2O$  and extd. with  
 $Et_2O$ , and the residue after evapn. of the  $Et_2O$ , was chroma-  
 tographed to yield 105 mg. (41%) I,  $b_4$  95-105°. I was  
 also obtained by the Kishner reduction of the *semicarbasone*

of III in 50% yield. I from the 2 equivalents of propene, had  
 $d_4$  0.8531, 0.3337; and  $d_4$  1.0621, 1.0628; the infrared  
 spectrum of I is given. LIV. The structure of lactaragu-  
 lone and lactaroviolin. František Šorm, Věra Benešová,  
 and Vlastimil Herout. *Ibid.* 18(6-61).—A new structure  
 for *lactarasulene* (from *Lactarius deliciosus*) (I).  $C_{10}H_8$  was  
 confirmed as 7-isopropenyl-1,4-dimethylazulene by partial  
 hydrogenation, which transformed I to *guiazulene* (II).  
*Lactarovolin* (III) also belongs to the guiazane (IV) type.  
 Ozonization of I showed the presence of methylene double  
 bond. Hydrogenation of I in  $Et_2O$  over Pd on C, deacti-  
 vated with quinoline vapors, gave II, *trinitrobenzene compd.*,  
 m. 151°. III, m. 87.5-8° (from petr. ether-C<sub>6</sub>H<sub>6</sub>) which  
 showed the presence of 1  $CH_3$  group, gave, by hydroge-  
 nation over  $PtO_2$  in  $AcOH$ , IV,  $C_{10}H_8$ ,  $d_4$  0.8810,  $n_D^{20}$  1.4811,  
 and an alc.,  $C_{10}H_8O$ ,  $b_4$  180-5° (bath temp.). Partial  
 hydrogenation over deactivated Pd gave dihydrolactarovilo-  
 lin,  $b_{44}$  130-8° (bath-temp.). Infrared spectra of I and  
 IV are given. LVI. Paper chromatography of azulenes.  
 Otto Knessl and Alice Vlastiborová. *Ibid.* 48, 212-16  
 (1964).—The analysis of triazulenes is based on dif-  
 ferent  $R_f$  values obtained by paper chromatography with  
 petr. ether as stationary and 35-70%  $H_3PO_4$  as mobile  
 phases. The  $R_f$  values with 35, 45, 50, 55, 60, 65, and 70%  
 $H_3PO_4$ , resp., are listed for *S*-guiazulene (0.0, 0.08, 0.30,  
0.43, 0.68, 0.87, 1.0), *selinazulene* (0.0, 0.02, 0.13, 0.23,  
0.47, 0.65, 1.0), *chamazulene* (0.03, 0.23, 0.47, 0.65, 0.79,  
0.90, 1.0), and values for 50 and 55%  $H_3PO_4$ , resp., are  
given for *Se*-guiazulene (0.66, 0.80), *isoguaiazulene* (0.50,  
0.72), and *lindazulene* (0.02, 0.08). The chromatograms  
were developed with  $Li_2O$  or dry  $NH_3$ . LVII. Identity of  
lindazulene with chamazulene. F. Šorm, V. Herout, and  
K. Takeda. *Ibid.* 281-3; *Collection Czechoslov. Chem. Com-  
muniq.* 19, 180-8 (1954) (in English).—Identity of lind-  
azulene (*C.A.* 44, 917; 48, 7710g) with chamazulene (1,4-  
dimethyl-7-ethylazulene) was proved by mixed m.p.s. of the  
trinitrobenzenes compds. and by comparison of visible and  
ultraviolet spectra. The partial synthesis of lindazulene  
(*Pharm. Bull. Japan* 1, 241 (1953)) is, at the same time,  
proof of the correct structure of chamazulene as proposed by  
Šorm, et al. (see 5th preceding abstr.). Dr. Hradilek

VLADIMÍR SYKORA, VLASTIMIL

HEROUT, et al.

Substances of *Artemesia absinthium* and isomeric w<sup>o</sup> crystalline prochamazulenogenen. Vlastimil Hrdou and František Šorm (Čech. Akad. Věd, Prague, Czech.). *Česk. Články* 47, 1011-52 (1953); *Collection Czechoslov. Chem. Článků*, 18, 854-69 (1953) (in English).—From the petr. ether ext. of *A. absinthium*, a hydroxylactone C<sub>17</sub>H<sub>20</sub>O has been isolated which is believed to be a compd. from which originate the chamazulenogenen substances (pro-chamazulenogenen) (I). Ext. of the dry drug (760 g.) with petr. ether gave 7.1% of an ext. contg. 1.18% chama-*sulene* (II). The ext. dissolved in 2 l. petr. ether extd. with 8 250 ml. portions of 50% EtOH, washed three times with 300 ml. 3% NaCl, dried and evapd. gave 42 g. of a waxy substance (fraction A). Acidification of the alk. washings with dil. H<sub>2</sub>SO<sub>4</sub> and extn. with Et<sub>2</sub>O gave 1.8 g. dark-green acids. 1.1 g. of the EtOH from the aq.-alc. exts. and extn. with Et<sub>2</sub>O yielded 10.8 g. (fraction B), contg. 5.44% II. Chromatography of fraction A (29 g.) gave 10 g. C<sub>21</sub>H<sub>30</sub>, m. p. 100° (after suid. 100°/0.1 mm.), waxy carotenoïd compds., (0.8 g.) ester with sapon. no. 97.6 (C<sub>21</sub>H<sub>30</sub>O<sub>2</sub> acid), a compd. m. 108° (from AcOEt and petr. ether), a mixt. of (3.55 g.) alcohols CuH<sub>20</sub>O and C<sub>21</sub>H<sub>20</sub>O (III), m. 74-5° (from AcOEt and Me<sub>2</sub>CO), a mixt. of 30 mg. of phytosterols, m. 139-41° (C<sub>21</sub>H<sub>30</sub>O) (acetyl-deriv., m. 117°, from EtOH), and palmitic acid, m. 61° (approx. 1 g.). Fraction B deposited 1.1 g. CuH<sub>20</sub>O, m. 165.5° (from C<sub>21</sub>H<sub>20</sub>-EtOH). Chromatography of the rest (9.4 g.) yielded 0.5 g. III, 180 mg. of a compd., m. 114°, 0.45 g. absinthin, m. 254-7°, and 850 mg. I, m. 129-32° (decompn.) (from C<sub>21</sub>H<sub>20</sub>) sol. in EtOAc, Me<sub>2</sub>CO, Et<sub>2</sub>O, and EtOH, slightly in sol. petr. ether, CS<sub>2</sub>, CCl<sub>4</sub>, H<sub>2</sub>O. Hydrogenation of I in AcOH over PtO<sub>2</sub> gave, after chromatography, C<sub>21</sub>H<sub>30</sub>O, b.p. 160-6° (bath temp.), CuH<sub>20</sub>O, b.p. 130° (bath temp.), and a compd. m. 108°. Sapon. of I with 5% alc. KOH under standard conditions yielded upon decarboxylation 18.3% II.

M. Hudlický

S.M., F.

Mitrović, C.; Milić, I.  
"Quinolizidine Alkaloids. II. Synthesis of Three Isomeric  $\beta$ -(-liperidyl)-  
Quinolizidines." p. 1993. (Chemical Listy. Vol. 47, No. 7, July 1953, Praha.)

See: Monthly List of East European Publications, Library of Congress, March 1953, Vol. 1, No. 3.

Schulz, P.; Neffall, J.; Hennig, R.  
"Terpenes. VI. The Isolation of Chamaulan; A Preparatory  
Concise Edition," p. 197. (Chemische Rundschau, Vol. 37, No. 7,  
July 1953, Braunschweig.)

See: Periodical List of Patent Applications, Library of Congress, April 1953, Vol. 3, No. 3.

SORM, F.

**Synthesis of *I*-asazinium[*cyclo-[0.3.3.3]*]undecane bromide.**

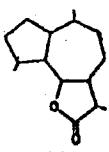
František Sorm and Jiří Beránek (Czech. Akad. Věd, Prague, Czechoslovakia). *Chem. Listy* 47, 1359-65 (1953).—An entirely sym. tricyclic compd. contg. a central quaternary N in the pos. ion, *I*-asazinium[*cyclo-[0.3.3.3]*]undecane (*I*), was prep'd. by two similar methods, both starting from  $O_2NC(CH_2CH_2CN)_2$  (*Ia*). *Ia*, m. 114°, prep'd. in 49% yield (Bruson and Reiner, *C.A.* 37, 1331!) (with [PhMe<sub>2</sub>N]<sub>2</sub>OEt as catalyst, gave by hydrolysis 77%  $O_2NC(CH_2CH_2CO_2H)_2$ , m. 180°, which yielded by azeotropic esterification (catalyst  $\beta$ -Me<sub>2</sub>CH<sub>2</sub>SO<sub>3</sub>H) 86.4%  $O_2NC(CH_2CH_2CO_2Et)_2$  (*II*), b.p. 155-80°, n<sub>D</sub><sup>20</sup> 1.4629. Hydrogenation of 36 g. *II* over 3 g. Raney Ni at 110° and 110 atm. gave, after 1 hr., 28.5 g. (89.7%) 5,5-bis( $\beta$ -carbethoxyethyl)-2-pyrrolidone (*III*), m. 46° (from Et<sub>2</sub>O), b.p. 201°. Heating *III* (10 g.) 5 hrs. at 205-10° and 12 mm. yielded 8.8 g. (78.8%) 8-( $\beta$ -carbethoxyethyl)-5-dioxopyrrolidine (*IV*), m. 103° (from EtOH or dioxane), b.p. 220-4°. A soln. of 8.3 g. *IV* in 130 ml. dioxane was dropped during 7 hrs. into a suspension of 4 g. LiAlH<sub>4</sub> in 50 ml. dioxane heated to 70-80°, the mixt. allowed to stand at room temp. overnight, decompr'd. with 5 ml. H<sub>2</sub>O, then with 12 ml. 20% NaOH, filtered, the ppt. washed with 100 ml. dioxane, and the filtrate evap'd. to give, on distn., 3 g. (51%) 8-( $\gamma$ -hydroxypropyl)pyrrolidines (*V*), b.p. 132-3°, d<sub>4</sub><sup>20</sup> 1.0354, n<sub>D</sub><sup>20</sup> 1.4958 (paraff., m. 146-7°), and 1.9 g. (29.2%) 2,2-bis( $\gamma$ -hydroxypropyl)pyrrolidine (*VI*), b.p. 172-6°, m. 76-7° (from AcOEt). Heating 2.3 g. *V* with 30 ml. 27% HBr in AcOH 11 hrs. at 100° (autoclave) yielded 4 g. (94%) HBr salt of 8-( $\gamma$ -bromopropyl)pyrrolidine

(*VII*), m. 133° (from Me<sub>2</sub>CO). Treating 2.3 g. *VII* in 60 ml. H<sub>2</sub>O with Ag<sub>2</sub>O prep'd. from 3.5 g. AgNO<sub>3</sub>, filtering off the AgBr, and mixing the soin, with picric acid gave 2.7 g. (96.5%) picrate of *I*, m. 318°. Mixing 1.7 g. *I* picrate in 10 ml. H<sub>2</sub>O with 12 ml. 48% HBr and removing the picric acid by ether extn. yielded 0.9 g. (87%) *I* bromide, m. above 380° (from Et<sub>2</sub>O-Me<sub>2</sub>CO). Adding 9.5 g. *III* in 130 ml. dioxane during 2 hrs. to 3.8 g. LiAlH<sub>4</sub> in 60 ml. dioxane at 70-80°, continuing the heating for 2 hrs., and decomprg. the mixt. with 5 ml. H<sub>2</sub>O and 13 ml. 20% NaOH yielded 5.1 g. (81.0%) *VI*, b.p. 176-7°. *VI* (4 g.) heated in an autoclave 14 hrs. at 103° with 50 ml. 31% HBr in AcOH yielded 7.3 g. (87%) HBr salt of 2,2-bis( $\gamma$ -bromopropyl)pyrrolidine (*VIII*), m. 95-6° (from AcOEt). Treating 6.3 g. *VIII* in 150 ml. H<sub>2</sub>O with Ag<sub>2</sub>O prep'd. from 11 g. AgNO<sub>3</sub> gave hydride of *I* which yielded, on distn., 58.5% 8-allipyrrolidined (IX), b.p. 191-3°, b.p. 72°, d<sub>4</sub><sup>20</sup> 0.9323, n<sub>D</sub><sup>20</sup> 1.4812 (picrate, m. 183°). *IX* was hydrogenated to 8-propylpyrrolidine, b.p. 100°, n<sub>D</sub><sup>20</sup> 1.4646 (picrate, m. 156-6°). *I* bromide crystallizes from EtOH in the hexagonal system. Also in *Coll. Czechoslov. Ch. a. Commun.* 19, 298-303 (1954) (in English).

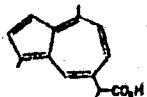


*CZECH*

*Chamazulene precursor from chamomile (*Matricaria chamomilla L.*). Z. Čekan, V. Herout, and F. Šorm. (Czech. Acad. Sci., Prague). Chemistry & Industry 1956, 604-6; cf. C.A. 49, 997c.—Cryst. I,  $C_{11}H_{16}O_4$ , m. 188-60° (decompn.), having 2 double bonds, an acetyl and hydroxyl group of unassigned position has been isolated in cryst. form from *Matricaria chamomilla L.* as a precursor of chamazulene (II). The ultraviolet absorption spectrum showed a max. at 243  $\mu$ , log E 4.32. On hydrogenation*



(I)



(III)

2.9 equiv. of H were absorbed and 2, probably stereoisomeric, lactones,  $C_{10}H_{16}O_4$ , m. 115.5° and 122-3°, were isolated. Steam distn. of I gave 70-75% II by the elimination of 3 HO groups and subsequent decarboxylation. III is proposed as an intermediate in this reaction (Jung and Wendler, C.A. 47, 10800g) and the name guaiaulenic acid is suggested. The name guaianolides is proposed for compds. contg. a lactone group on a guaiane skeleton. The antiphlogistic activity of I has been found to be at least equal to that of II. M. M. Bender

*R. J. G.*

*SOKA, F.*

Presence of 1,7-dimethyl-4-isopropyl-decahydronaphthalene in the carbon skeleton of Interpetine. Form. M. Holub, and V. Hrout (Czech. Acad. Sci., Prague, Chem. Industry) 1954, 686. The C skeleton of Interpetine is established by treating dehydroisobenzyl with HI and subsequent dehydrogenation with S, which yielded a naphthalenic hydrocarbon. The consts. of the free hydrocarbon, the picrate, and styphnate correspond to those of 1,7-dimethyl-4-isopropyl-naphthalene and its appropriate derivs.

George B. Peat

*M. S. H.*

SORRY, F.

The structure of lactoviolin. P. Form, V. Benešová, CH  
V. Kuncík, V. Šebek, I. Doležal, V. Herout, and J. Šloher  
(Czech. Acad. Sci., Prague). Chemistry & Industry 1954,  
1611-18.—The structure 1-formyl-4-methyl-7-isopropenyl-  
azulene (1) (Heilbronner, *Chemie* 8, 97 (1954); Pfitzner  
*et al.*, *C.A.* 49, 147264) for lactoviolin is further supported  
by proof (*C.A.* 48, 127068) of the guaiane (perhydroguaiar-  
ene) skeleton. Comparison of the polarographic reduction  
potentials of lactoviolin and its dimethoxy-deriv. excludes  
1,4-dimethyl-7-( $\beta$ -acrylaldehyde)azulene. Reaction of lac-  
toviolin with MeMgI gives the unstable 1-methylmethylol-  
4-methyl-7-isopropenylazulene, which on hydrogenation  
(Pd-C; deactivated with quinoline) affords 1-ethyl-4-methyl-  
7-isopropylazulene, *trinitrobenzene addn. compd.*,  $C_9H_{12}O_3N_3$ ,  
m. 111°, not depressed with synthetic material, infrared  
spectra of the natural and synthetic azulenes are identical.  
The isomer, 4-ethyl-1-methyl-7-isopropylazulene, *trinitroben-  
zene adduct*,  $C_{10}H_{14}O_3N_3$ , m. 117°, is depressed with the deriv.  
from lactoviolin. The azulenes were synthesized by  
methods previously reported (*C.A.* 47, 4313c and 4314a).  
Chloroazulene has recently been synthesized by these methods.  
F. R. Mumford

(6)

R.F.  
M.G.T.

LAUCIKOVA, O.; JAKUBOVIC, A.; KEIL, B.; SORM, F.

Viruses I; isolation and chemical properties of Rous sarcoma.  
Chekh. biol. 3 no.5:298-307 Nov 54.

1. Institut organicheskoy khimii ChSAN, organicheskaya biokhimiya,  
Praga.

(VIRUSES,  
Rous sarcoma virus, isolation & chem.)

(NEOPLASMS, experimental,  
Rous sarcoma virus, isolation & chem.)

(SARCOMA, experimental,  
Rous sarcoma virus, isolation & chem.)

SORM, F.; MALEK, I.

Flow cultivation of yeast ferment. p. 379  
CESKOSLOVENSKA BIOLOGIE, Vol. 3, No. 6, Nov. 1954

SO: Monthly List of East European Accessions, (EEAL), LC, Vol. 4, No. 5 Sept. 1955  
Uncl.

SHORM, F.

USSR/ Scientific Organization - Czechoslovakia

Card 1/1 : Pub. 124 - 12/24

Authors : Shorm, F., Academician, Chief Secretary of the Acad. of Sc.  
Czechoslovakia

Title : The first year of the Czechoslovakian Academy of Sciences

Periodical : Vest. AN SSSR 9, 65-69, Sep 1954

Abstract : Progress report on the activities of the Czechoslovakian Academy of Sciences during its first year of existence is presented.

Institution : Academy of Sciences, Prague, Czechoslovakia

Submitted : ...

FAJKOS, J.; SORM, F.

Steroids. Part 7. Synthesis of  $3\beta$ -hydroxyandrosta-7,16-diene-16-carbo-  
xylic acid [in Russian with summary in English]. Sbor.Chekh.khim.rab. 19  
no.1:91-97 P 154. (MLRA 7:6)

1. Department of Natural Products, Institut of Organic Chemistry Czechoslovak Academy of Science, Prague. (Acids, Organic) (Androstadiene)

RATUSKY, J.; SORM, F.

Alkaloids derived from quinolizine. Part 3. Synthesis of three stereoisomeric  
3-( $\alpha$ -piperidyl)-quinolizidines [in Russian with summary in English].  
Sbor.Chekh.khim.rab. 19 no.1:107-117 F '54. (MLRA 7:6)

1. Department of Organic Synthesis, Institute of Organic Chemistry,  
Czechoslovak Academy of Science, Prague. (Alkaloids) (Quinolizidine)

SYKORA, V.; HEROUT, V.; PLIVA, J.; SORM, F.

Terpenes. Part 50. Contribution to the constitution of elemol [in English  
with summary in Russian]. Sbor.Chekh.khim.rab. 19 no.1:124-134 F '54.  
(MLRA 7:6)

1. Department of Natural Products, Institute of Organic Chemistry,  
Czechoslovak Academy of Science, Prague. (Elemol)

SORM, F.

SORM, F.; HOLUB, M.; HEROUT, V.

Terpenes. Part 52. Constitution of laserpitine [in German with summary in Russian]. Sbor.Chekh.khim.rab. 19 no.1:135-140 F '54. (MLRA 7:6)

1. Otdeleniye prirodnykh veshchestv, Institut organicheskoy khimii  
Chekhoslovatskoy Akademii nauk, Praga. (Laserpitine)

Smit, F.; van der, H.; Hart, R.

"Amino Acids and Peptides. IX. Constitution of the Peptide Phalloidine. II."  
P. 153, (COLLECTION OF CZECHOSLOVAK CHEMICAL COMMUNICATIONS. SBORNIK CHEKOSLOVAT-  
SKIKH KHIMICHESKIKH RABOT, Vol. 19, No. 1, Feb. 1954, Praha, Czechoslovakia)

SO: Monthly List of East European Accessions, (EEL), 1C, Vol. 4  
No. 5, May 1955, Uncl.

SORM, F.

MELOUN, B.; KEIL, B.; SORM, F.

Amino acids and peptides. Part 9. Constitution of the peptide phalloidine; part 2 [in German with summary in Russian]. Sbor.Chekh.khim.rab. 19 no.1:153-161 F '54. (MLRA 7:6)

1. Otdeleniye organicheskoy biokhimii, Institut organicheskoy khimii Chekhoslovatskoy Akademii nauk, Praga. (Phalloidine)

*SOKA E.*

RYCHLIK, I.; SOBM, P.

Proteins. Part 20. Specificity of dinitrophenyl derivatives of  $\alpha$ -chymotrypsin and of trypsin [in Russian with summary in English]. Sbor.Chekh. khim.rab. 19 no.1:162-166 P '54. (MIRA 7:6)

1. Department of Biochemistry, Institute of Organic Chemistry, Czechoslovak Academy of Science, Prague. (Trypsin)

Sokrm, Frantisek

Inhibitory effect of chloramphenicol on the formation of  
some enzyme systems of *Escherichia coli*. František Sokrm  
and Dezider Grünberger (Czech. Acad. Sci., Prague). Col-  
lection Czechoslov. Chem. Commun. 19, 167-73 (1954) (in  
English). See C.A. 48, 4044b. E. J. C.

SORM, F.

SORM, F.; HEROUT, V.; TAKEDA, K.

Terpenes, Part 54. Identity of lindazulene and chamaulene [in English with summary in Russian]. Sbor.Chekh.khim.rab. 19 no.1:186-188 F '54.  
(MLRA 7:6)

1. Department of Natural Substances, Institute of Organic Chemistry,  
Czechoslovak Academy of Science, Prague and Research Laboratory,  
Shionogi & Co., Imafuku, Amagasaki, Hyogo-ken, Japan.  
(Lindazulene) (Chamaulene)

SORM, F.; BERANEK, J.

Synthesis of 1-azonia-tricyclo-(3,3,3,0)-undecane bromide [in English  
with summary in Russian]. Sbor.Chekh.khim.rab. 19 no.2:298-304 Ap '54.  
(MLRA 7:6)

1. Department of Organic Synthesis, Institute of Organic Chemistry.  
Czechoslovak Academy of Science, Prague.  
(Undecane bromides)

SORM, F.  
SICHER, J.; SVOBODA, M.; FARKAS, J.; SORM, F.

Studies in the chloramphenicol series. Part 7. The side reactions  
in the reduction of dehydrochloramphenicol [in English with summary in  
Russian]. Sbor.Chekh.khim.rab. 19 no.2:317-329 Ap '54. (MLRA 7:6)

1. Department of Organic Synthesis, Institute of Organic Chemistry,  
Czechoslovak Academy of Science, Prague. (Chloromycetin)

SCRN FRANTISEK

CZECH

*Odeanolide alkaloids. IV. A new synthesis of (±)-lupinine, (±)-segetoline, (±)-J-lupinine, and (±)-epilupinine.* Josef Ratajek and František Šorm (Czech. Acad. Polv. Prague). Collection Czechoslovak. Chem. Comm., 19, 340-8 (1964) (in Russian). — See C.A. 49, 836a.

H. J. C.

R. H.

SORM, F.

FAJKOS, J.; SORM, F.

On steroids. Part 9. Synthesis of  $\beta$ -hydroxy-16-ketoandrostane and of  
3,16-diketoandrostenes-(4) [with summary in English]. Sbor.Chekh.khim.rab.  
19 no.2:349-356 Ap '54. (MLRA 7:6)

1. Department of Natural Products, Institute of Organic Chemistry,  
Czechoslovak Academy of Science, Prague.  
(Androgens)

Sorm, František

> Terpenes. LIV. The structure of lactarazulene and  
lactaroviolin. František Sorm, Věra Benešová, and Vlasti-  
ná Šimková (Czech. Acad. Sci., Prague). Collection Czecho-  
slov. Chem. Commun. 19, 351-63 (1954) (in German).—See  
C.A., 48, 12708e.  
E. L. C.

RA  
Met

**C Z E C U**

Steroids. X. Dimethylamino derivatives of allo-  
pregnane. Jiri Laska, Václav Černý, and František Šorin  
(Inst. Org. Chem., Czech. Acad. Sci., Prague). *Czechoslovak. Chem. Commun.* 19, 651-8 (1954) (in English).—  
See C.A. 49, 368d. E. J. C.

*B1*

*sekýr, karel řehoř*

**C Z E C H**

*V Terpenes. LIII. Synthesis of 2,8,8-trimethylbicyclo-(3,2,0)-octane: proof of the constitution of  $\beta$ -caryophyllene.*  
Ladislav Doležil and František Šorm. (Czech. Acad. Sci., Prague). *Collection Czechoslov. Chem. Commun.* IV, 609-615 (1964) (in English).—See *C.A.* 68, 12707*i*. B. J. C.

*M. Šorm*

SKM, FLAWED 5

CZECH

Vorpanec, LV. Synthesis of elemene (1-methyl-1-  
ethyl-2,4-disisopropylcyclohexane). Vladimír Štoka, I.H.  
Černý, Vlastimil Herout, and František Šorm (Czech.  
Acad. Sci., Prague). Collection Czechoslov. Chem. Com.  
19, 565-9 (1964) (in English).—See C.A. 49, 1655i.

R. J. C.

RESEARCH

..., F., and others.

"Terpenes. LVIII. Total Synthesis of 1,1,4,8-Tetramethylcycloundecane; Proof of the Eleven-Membered Ring in Humulene. In English." p. 570, (COLLECTION OF CZECHOSLOVAK CHEMICAL COMMUNICATIONS. SLOVNIK CHEKHOVSKO-ATOMSKIH KHIMICHESKIKH RABOT, Vol. 19, No. 3, June 1954, Praha, Czechoslovakia)

cc: Monthly List of East European Accessions, (EEL), LC, Vol. 4  
No. 5, May 1955, Uncl.

...M., ...M.

"Effect of Aconitine on the Metabolism of Brain Tissue." p. 580,  
SLOVAKSKA CHIMICKA OBRAZOVATELNA  
(COLLECTION OF CZECHOSLOVAK CHEMICAL COMMUNICATIONS. SLOVAKIA CHIMICOVATELNA  
AKADEMICKA REDIT, Vol. 19, No. 3, June 1954, Praha, Czechoslovakia)

SI: Monthly List of East European Accessions, (EMAL), LC, Vol. 4  
No. 5, May 1955, Uncl.

FAJkus, J.

Hrubá, J. Preparation of polymeric hydroxy- $\alpha$ -carboxylic  
acid. (Collection of Czechoslovak Chemical Communication. Praha. Vol. 19,  
no. 4, Aug. 1954) /East  
Europe Scientific Information (EEI), 16, Vol. 1, No. 1,  
June 1955, p. 1.

CEKOV, Z.; VESCHT, V.

(Collection of Czechoslovak Chemical Communication. Praha. Vol. 19,  
no. 4, Aug. 1954) East  
V: Chemical Abstracts (C.A.), 38, Vol. 1, no. 6,  
Aug. 1954.

$\Sigma(Km)$  /

VETL, V. J. KVASLOVA, V.

(Collection of Czechoslovak Chemical Communication. Praha. Vol. 19, no. 4, Aug. 1954)

**APPROVED FOR RELEASE: 08/25/2000**

CIA-RDP86-00513R001652420014-6"

Sorm, Fr.

**EXC II**

Proteins. XXV. Chemical structure of proteins. František Sorm. Collection Czechoslov. Chem. Commun. 19, 1000-1014 (in Russian).—See C.A. 48, 13747f. XXVI. Photometric analysis of protein hydrolyzates. Boživoj Keil. Ibid. 1000-17.—See C.A. 48, 13549i. XXVII. Comparative study of the acidic peptide fractions from partial hydrolyzates of chymotrypsinogen and trypsinogen. Boživoj Keil and František Sorm. Ibid. 1018-31.—See C.A. 48, 13747f.

E. J. C.

RE/POX

MILAN; KELL, R.

Grajeda, MM. Comparative study of the yields of the fractions from  
catalytic hydrolysis of the lignin and hemicellulose. p. 210 (Collection of Czechoslovak Chemical Fast Communication. Praha. Vol. 19, no. 5, Oct. 1954)  
Also published in: Acta Academiae (CZ), 1954, Vol. 1, No. 6,  
pp. 101-103, 1954.

SORM, F.

CZECH

6190\* Mechanism of Antibiotic Action. O mekanizme  
dействия антибиотиков. II. Specific Action of *d*-Chloram-  
phenicol on the Development of Some Seedling Plants. Spetsi-  
ficheskoe delstvie *d*-khloramfenikola na razvitiye rostkov  
rastenii. (Russian.) F. Sorm, M. Zelinkova, and Z. Sormova.  
*Collection of Czechoslovak Chemical Communications*, v. 19,  
no. 6, Dec. 1954, p. 1324-1329.

Seeds with different reserves of protein, starch, and oil gave  
different responses to the inhibitory effect. Graphs. 9 ref.

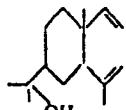
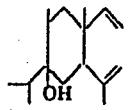
SORM, F., akademik.

Report of the year's activity of the Academy of Sciences of Czechoslovakia. Vest. AN SSSR 24 no.9:65-69 S '54. (MLRA 7:9)

1. Glavnnyy uchenyy sekretar' Chekhoslovatskoy Akademii nauk.  
(Academy of Sciences of Czechoslovakia)

*Sorm, Frantisek*

Terpenes. LV. Synthesis of elemene (1-methyl-1-ethyl-2,4-diisopropyl cyclohexane). Vladimir Šikora, Jitka Černá, Vlastimil Herout, and František Sorm (Csl. akad. ved, Prague, Czech.). *Chem. Listy* 48, 10-14 (1954); cf. preceding abstr.—Synthetic 1-methyl-1-ethyl-2,4-diisopropyl cyclohexane (I) is identical with elemene, a reduction product of elemol. However, 1,5-diisopropyl-1-methyl-2-ethylcyclohexanol (II) is not identical with tetrahydroelemol. One of the three formulas is suggested for elemol.



To a suspension of dry MeONa (prepd. from 23 g. Na) in 200 ml. C<sub>6</sub>H<sub>6</sub>, was added 75 g. HCO<sub>2</sub>Et in 200 ml. C<sub>6</sub>H<sub>6</sub>, and to the ice-cooled mixt. was added a soln. of 51.5 g. carvomenthone (b<sub>1</sub> 94-4.5°, n<sub>D</sub><sup>20</sup> 1.4513) in 300 ml. C<sub>6</sub>H<sub>6</sub>. After 48 hrs. at room temp. under N atm., the mixt. was decomposed with H<sub>2</sub>O, the C<sub>6</sub>H<sub>6</sub> layer repeatedly extd. with 7% NaOH, the alk. ext. was extd. with Et<sub>2</sub>O, then acidified with HCl (1:1) to pH 8, and extd. again with Et<sub>2</sub>O to give 47 g. (83%) 2-methyl-5-isopropyl-6-formylcyclohexanone (formylcarvomenthone) (III), b<sub>1</sub> 122-2.5°. Etherification of III

(47 g.) with 38 g. Li-BuOH yielded 49 g. (80%) 2-methyl-5-isopropyl-6-(isobutylmethylenecyclohexanone (IV), b<sub>1</sub> 112-16°. To ethylate IV, KNH<sub>2</sub> prepd. from 16 g. K in 160 ml. liq. NH<sub>3</sub> with 0.1 g. Fe(NO<sub>3</sub>)<sub>3</sub>, was added to 500 ml. boiling Et<sub>2</sub>O, the NH<sub>3</sub> was driven off under N atm., 44.7 g. IV in 200 ml. Et<sub>2</sub>O was added during 2 hrs. after 1.5 hrs. boiling, in the course of 2 hrs., 90 g. RII in 150 ml. Et<sub>2</sub>O was added, and the mixt. refluxed 12 hrs., treated with H<sub>2</sub>O, the aq. layer extd. with Et<sub>2</sub>O, the ext. washed with 5% KOH, H<sub>2</sub>O, dried, the Et<sub>2</sub>O evapd., and the residue mixed with 250 ml. *M* methanolic FeCl<sub>3</sub>. Treating the Fe complex with 400 ml. HCl (1:1), extg. the mixt. with Et<sub>2</sub>O, washing the ext. with dil. HCl and H<sub>2</sub>O, extg. the ether soln. repeatedly with 5% KOH, and steam distg. the alk. soln. yielded 17.5 g. (61.2%) 2-methyl-2-ethyl-5-isopropylcyclohexanone (V), pure, b<sub>1</sub> 113-3.5° (18.5 g.); semicarbazone, m. 111.5-12.5° (from aq. MeOH). Adding 1.82 g. V to a soln. of iso-PrLi (prepd. from 0.7 g. Li and 10 ml. iso-PrCl), and heating the mixt. 1.5 hrs. gave 2.1 g. 1,5-diisopropyl-2-methyl-2-ethylcyclohexanol (VI), b<sub>1</sub> 141-5°. Dehydration of 1.5 g. VI by heating 40 min. on the steam bath with fivefold excess of 80% HCO<sub>2</sub>H, chromatography, and hydrogenation of the petr. ether fraction (0.9 g., b<sub>1</sub> 112-13°) over PtO<sub>2</sub>, gave 1-methyl-1-ethyl-2,4-diisopropylcyclohexane (VII), d<sub>25</sub> 0.8480, n<sub>D</sub><sup>20</sup> 1.4638. Infrared spectra of VII and of elemene obtained by total reduction of elemol are identical.

M. Hudlický

SCAM, Frantisek

Chemical Abst.  
Vol. 48 No. 9  
May 10, 1954  
Biological Chemistry

①  
Effect of aconitine on the metabolism of brain tissue.  
František Šorm and Zdenka Beránková (Czech. akad. věd, Prague, Czech.). Chem. Listy 48, 80-8 (1954).—The influence of aconitine upon metabolism of the gray matter was followed. Low concns. of aconitine (0.02-0.2 mg. %) stimulate considerably the metabolism. Higher concns. first stimulate, then inhibit metabolism. The stimulation which is specific for the brain tissue is ascribed to the activation of the O bond.  
M. Hudlický

~~PRINTED~~ - GZL-01  
SORM, FRANTÍČEK

The reaction of ketene with hydrogen cyanide <sup>1H</sup>  
Smrt and František Sorm (Czech. akad. věd, Prague,  
Czech.), Chem. Listy 48, 217-220 (1954).—In the presence  
of basic catalysts, CH<sub>2</sub>:CO and HCN form a mixt. of CH<sub>2</sub>:C-  
C(CN)OAc (I) and MeC(CN)OAc (II). The yield of I is  
higher at -70° to -60°. The best yields of I were ob-  
tained with PhNMe<sub>2</sub>, (71%), Et<sub>3</sub>NH (61.5%), and EtONa  
(54%) as catalysts. The structure of I, formed as a primary  
product, was proved by its transformation to BrCH<sub>2</sub>CH-  
(CN)OAc (III) and BrCH<sub>2</sub>CH(OH)CO<sub>2</sub>Et (IV). CH<sub>2</sub>:CO  
(1.6 moles) passed 2 hrs. through a soln. of 14.5 g. anhyd.  
HCN in 60 ml. Et<sub>2</sub>O contg. 0.6 ml. Et<sub>3</sub>NH and cooled to  
-60°, and the mixt. allowed to stand 2 hrs. at room temp.  
and fractionated *in vacuo* gave 34.5 g. (61.5%) I, b.p. 62-4°.  
To prep. II, 29 g. HCN in 100 ml. Et<sub>2</sub>O, 25 ml. Ac<sub>2</sub>O, and 0.6  
ml. N(CH<sub>2</sub>CH<sub>2</sub>OH)<sub>2</sub> was treated with 2 moles CH<sub>2</sub>:CO at  
-5° during 4 hrs.; distn. *in vacuo* yielded 67 g. (95%) II,  
b.p. 105-7°, m. 70°. Heating in an autoclave 5.5 g. I 6 hrs.  
at 100° with 12.7 g. 81% soln. of HBr in AcOH gave, by  
vacuum distn., 5.5 g. (57%) III, b.p. 105°, n<sub>D</sub><sup>20</sup> 1.4798. Re-  
fluxing 3 g. III with 10 ml. 48% aq. HBr 4 hrs., evap. the  
soln. *in vacuo*, refluxing the residue with 50 ml. EtOH and  
50 ml. C<sub>6</sub>H<sub>6</sub> in the presence of 0.1 g. salicosalicylic acid 6 hrs.,  
dilg. the soln. with 10 ml. H<sub>2</sub>O, and extg. the mixt. with 16  
ml. Et<sub>2</sub>O gave 1.55 g. IV, m. 41°, b.p. 97°. I (5.5 g.) in 10  
ml. Et<sub>2</sub>O added with cooling to 8.5 g. C<sub>6</sub>H<sub>5</sub>N in 10 ml. Et<sub>2</sub>O  
gave, by vacuum distn., 11.8 g. (93%) AcNC<sub>6</sub>H<sub>5</sub>. b.p. 100°.  
Heating 3 g. I in the presence of 30 mg. Br<sub>2</sub>O<sub>2</sub> 80 hrs. at  
50-60° gave a glassy polymer, swelling in Me<sub>2</sub>CO, and  
polymerized to a rubberlike mass. M. Hudlický

SORM, F.; HEROUT, V.; TAKEDA, K.

"Terpenes. LVII. Identity of Linderazulene with Chamazulene", P. 281,  
(CHEMICKE LISTY, Vol. 48, No. 2, Feb. 1954, Praha, Czechoslovakia)

SO: Monthly List of East European Accessions, (EEAL), LC, Vol. 3, No. 12,  
Dec. 1954, Uncl.

SČRK, FRANTIŠEK

## C Z E C H

Ternes, LVIII. Total syntheses of 1,1,4,8-tetramethylcycloundecane (humulane). Proof of the eleven-membered ring in humulene. František Sčrk, Milan Streibl, Václav Jarolím, Ladislav Novák, Jiří Dolník, and Vlastimil Herout (Ustav Organické Chemie, CSAV, Praha, Czechoslovakia). *Collection Czechoslov. Chem. Listy*, 48, 873-83 (1953); *Collection Czechoslov. Chem. Commun.*, 19, 570-80 (1954) (in English); cf. *C.A.* 48, 197034.—1,1,4,8-Tetramethylcycloundecane (**I**) was synthesized by 2 different ways; a 3rd possible way was abandoned as unsuitable at the stage of HO<sub>2</sub>CCHMe(CH<sub>3</sub>)CMe(CH<sub>3</sub>)<sub>2</sub>CH(MeCO<sub>2</sub>H) (**II**). The identity of **I** was proved by d., n, and infrared spectra. BrO<sub>2</sub>CCH<sub>2</sub>CMe<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>Et (1.2 g.) in 10 ml. Et<sub>2</sub>O was dropped into 30 ml. Br<sub>2</sub>O contg. 1 g. LiAlH<sub>4</sub>, and the mixt. refluxed 1 hr., decompr., with H<sub>2</sub>SO<sub>4</sub> and extd. with Et<sub>2</sub>O to give 0.78 g. (80%) HO(CH<sub>2</sub>)<sub>2</sub>CMe<sub>2</sub>CH<sub>2</sub>OH (**III**), b.p. 150°. **III** (15 g.) add. with HBr at 95-133° gave 21.8 g. (82%) Br(CH<sub>2</sub>)<sub>2</sub>CMe<sub>2</sub>CH<sub>2</sub>Br (**IV**), b.p. 124-3°. The Na salt of MeCH<sub>2</sub>(CO<sub>2</sub>Et)<sub>2</sub> (**V**) prep'd. from 7 g. **V** with 0.92 g. Na dust in 30 ml. PhMe, treated in an autoclave 3 hrs. at 170° with 5.6 g. **IV**, gave 5 g. (37%) of the product of condensation of **IV** w/ 1 mole of **V**, b.p. 133-40° and 1.5 g. (16%) (EtO<sub>2</sub>C)CMe<sub>2</sub>CH<sub>2</sub>CMe<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>Et (**VI**), b.p. 170°. Hydrolysis of **VI** with KOH in MeOH gave 67% (HO)<sub>2</sub>CMe<sub>2</sub>CH<sub>2</sub>CMe<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub>Et, m. 160-5° (decompn.). Decarboxylation of the acid at 170-80°/20 mm. yielded 74% glassy **II**, b.p. 160-4°; di-Me ester (with Cl<sub>2</sub>N, in 82% yield), b.p. 115°. Me<sub>2</sub>C(CH<sub>2</sub>CO<sub>2</sub>Et)<sub>2</sub> (74 g.) in 100 ml. Et<sub>2</sub>O added in 30 min. to 13 g. LiAlH<sub>4</sub> in 600 ml. Et<sub>2</sub>O, the mixt. refluxed 30 min., decomp'd. with wet Et<sub>2</sub>O, H<sub>2</sub>O, and HCl, and repeatedly extd. with 4 l. Et<sub>2</sub>O gave 36 g. (80%) Me<sub>2</sub>C(CH<sub>2</sub>OH)<sub>2</sub> (**VII**), b.p. 140°. **VII** was transformed to 33% Me<sub>2</sub>C(CH<sub>2</sub>CH<sub>2</sub>Br)

SCHE, F.; SEKUT, V.; KETI, C.

"Terpenes. II. Composition of Juniper Oil", p. 590, (CHEMISTRY,  
Vol. 18, No. 4, April 1954, Praha, Czech.)

SC: Monthly List of East European Accessions (EEAI), 1C, Vol. 4, No. 3,  
March 1955, Urd.

SORM, F.

Proteins. XIV. Substitution of  $\epsilon$ -amino groups of lysine in the molecule of chymotrypsinogen by the reaction with dinitrofluorobenzene. Věra Kneslová, Bohumil Kell and František Sorm (Ústav Org. Chem., ČSAV, Praha, Czechoslovakia); *J. Pol. Sci.*, 18, 699-801 (1954); cf. *C.A.* 48, 64788.—By carrying out the reaction of 2,4-dinitrofluorobenzene (**I**) with chymotrypsinogen in a soln. of NaHCO<sub>3</sub> and 1% Me<sub>2</sub>N, under certain conditions all of the  $\epsilon$ -amino groups in lysine reacted with **I**. The importance of pH in the reaction is stressed. M. Hudlický

SORM, Frantisek

CZECH

*Reactions of ketene. III. The reaction of ketene with acid chlorides.* Jiri Berknek, Jiri Souto, and Prantsek  
Sofia (Czech. Akad. věd, Prague). *Chem. Listy* 48, 679.

(Received April 17, 1954) — Ketene (I) reacts with acid chlorides having next to the COCl group a  $\text{C}_2\text{H}_5$  group like  $\text{CH}_2\text{COCl}$ ,  $\text{CCl}_2\text{COCl}$ ,  $\text{CO}_2\text{Et}$ ,  $\text{CHCl}_2\text{COCl}$ ,  $\text{CO}_2\text{Et}$ , and  $\text{CH}_2\text{Ph}_2$ , to give primarily the corresponding aceto-acetyl chlorides. The ease of the reaction drops in the order given. Passing I (0.1 mole/lit.) into a soln. contg. 11 g.  $\text{CH}_2\text{COCl}$  in 50 ml.  $\text{CHCl}_3$  3.5 hrs. at  $-5^{\circ}\text{C}$ , esterifying the mixt. with 26 ml.  $\text{EtOH}$ , and distg. the product *in toto* yielded 15.3 g.  $\text{CO}(\text{CH}_2\text{COCl})_2$ , m. 105–107°, b.p. 92°, d<sub>4</sub> 1.113, n<sub>D</sub> 1.4426; *semicarbazone*, m. 90°. Treating 51.5 g.  $\text{CCl}_2\text{COCl}$  (II) in 50 ml.  $\text{CHCl}_3$  with I (0.1 mole/lit.) 3 hrs. at 13°, adding to the mixt. 50 ml. abs.  $\text{EtOH}$ , and distg. the mixt. *in toto* gave 19.5 g.  $\text{CCl}_2\text{COEt}$ , and 37 g. of a solid which yielded 17.1 g.  $\text{CH}_2\text{COCl}_2$ .

$\text{CH}_2\text{COCl}_2$  (III), m. 191° (decompn.) (from  $\text{CaH}_2$ ).

Fractionation of the mother liquors *in toto* gave 13.5 g.  $\text{CCl}_2\text{COCl}_2\text{CO}_2\text{Et}$  (IV), b.p. 100–102°. Solv. gave 22.3 g. II in 50 ml.  $\text{CHCl}_3$  1.5 hrs. at  $-5^{\circ}\text{C}$  with 1 (0.1 mole per lit.), esterifying the mixt. with 21 ml.  $\text{MeOH}$ , and distg. off the solvent and 12 g.  $\text{CCl}_2\text{CO}_2\text{Me}$ , b.p. 48°, gave 5.1 g. III and 1.35 g.  $\text{CCl}_2\text{COCl}_2\text{CO}_2\text{Me}$ , b.p. 90–92°. Treatment of 18.2 g. II in 20 ml.  $\text{CHCl}_3$  with 0.1 mole of I at 15° gave, after stripping off the solvent *in toto*, 6.2 g.  $\text{CCl}_2\text{COCl}_2\text{COCl}_2\text{CO}_2\text{Et}$ , unstable and hygroscopic crystals, m. 67–72°, giving III on melting. Passing 0.8 mole I into a soln. of 12.5 g. (IV) in 20 ml.  $\text{CHCl}_3$  at

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*Jiri Beranek*

(60-5° and distg. off *in vacuo* at 0° the unreacted ( $\text{COCl}$ )<sub>2</sub> (5.2 g., 41%), esterifying the residue by refluxing 30 min. with 20 ml. EtOH, and distg. the mixt. *in vacuo* gave 8.2 g.  $\text{Et}_2\text{COOCCH}_2\text{CO}_2\text{Et}$  (IV),  $b_1$  107-113°,  $b_2$ , 83°,  $d_{40}^{25}$  1.1324,  $n_{D}^{20}$  1.4385; *semicarbazone*, m. 162°. Satg. a soln. of 13.0 g.  $\text{CICOOC}_2\text{Et}$  in 20 ml.  $\text{CHCl}_3$  with 0.4 mole I at 15°, refluxing the mixt. 30 min. with 20 ml. abs. EtOH, and stripping off the solvents gave 2.2 g.  $\text{Et}_2\text{CC:CH-CO}_2\text{Et}$

$\text{CH}_2\text{CO}_2\text{Et}$ , m. 165° (from  $\text{CaH-EtOH}$  10:1), 3.15 g.  $(\text{CO}_2\text{Et})_2$ ,  $b_1$  72°, and 5.5 g. (20%) IV,  $b_1$  111-12°. Satg. a boiling soln. of 14.5 g.  $\text{CHCl}_3\text{COCl}$  in 20 ml.  $\text{CHCl}_3$  with 0.4 mole I and esterifying the mixt. with 20 ml. EtOH gave 9 g.  $\text{CHCl}_3\text{CO}_2\text{Et}$ ,  $b_1$  50°, and 2.5 g. (13%)  $\text{CHCl}_3\text{COCH}_2\text{CO}_2\text{Et}$ ,  $b_1$  104-5°,  $n_{D}^{20}$  1.4054. The reaction of I with AcCOCl (6 g.) yielded 4.05 g.  $\text{AcCO}_2\text{Et}$ ,  $b_1$  50-5°, and 1.12 g.  $\text{AcCOCH}_2\text{CO}_2\text{Et}$ ,  $b_1$  80-8°,  $b_2$  74-5°. Satg. the boiling soln. of 15.4 g.  $\text{PhCH}_2\text{COCl}$  in 20 ml. PhCl with 0.4 mole I, and esterifying the mixt. with 20 ml. EtOH yielded 14.8 g.  $\text{PhCH}_2\text{CO}_2\text{Et}$ ,  $b_1$  101-8°, and 2.02 g.  $\text{PhCH}_2\text{COCH}_2\text{CO}_2\text{Et}$ ,  $b_1$  180-6°,  $b_2$  144°. Also in Collection Czechoslov. Chem. Commun. 19, 1231-7(1954) (in German). M. Hudlicka

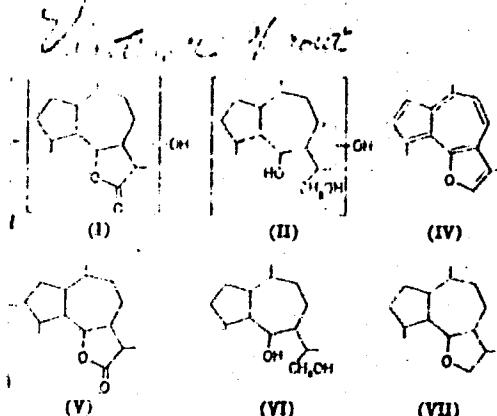
SČRM, FRANTISEK

CZECH

Terpenes. LXI. Constitution of prochamazulenogen C  
natural precursor of chamazulene in *Artemisia absinthium* ff ② 1/3

Vlastimil Hercout and František Šorm (Czech. akad. věd, Prague). *Chem. Listy* 48, 706-11; Collection Czechoslov. Chem. Communis. 19, 792-7 (1954) (in English); cf. *C.A.* 49, 4585d.—Prochamazulenogen C (2 double bonds) (*C.A.* 49, 097c), m. 133-5° (from EtOH),  $[\alpha]_D^{25} -49^\circ$ , reduced with LiAlH<sub>4</sub> in Et<sub>2</sub>O, gave a glassy product, a twice unsaturated triol (II), the dehydration of which by 00% HCO<sub>2</sub>H, by KHSO<sub>4</sub>, or by  $\beta$ -Me-C<sub>6</sub>H<sub>5</sub>SO<sub>3</sub>H gave only slight amt. of an azulene. Dehydrogenation of 200 mg. II by heating with 20 mg. S 30 min. at 180-195° yielded by petr. ether elution 2 g. guainzulene (III), and by C<sub>6</sub>H<sub>6</sub> elution, 45 mg. of a new blue azulene, artemazulene, C<sub>14</sub>H<sub>16</sub>O (IV) (infrared, visible, and ultraviolet spectra given); C<sub>6</sub>H<sub>5</sub>(NO<sub>2</sub>)<sub>2</sub> compd., m. 102° (from EtOH). Reduction of the mother liquors of prochamazulenogen (loc. cit.) with LiAlH<sub>4</sub>, and dehydrogenation with S yielded, besides small amts. of chamazulene and III, IV and a violet azulene C<sub>14</sub>H<sub>16</sub>O; C<sub>14</sub>H<sub>16</sub>(NO<sub>2</sub>)<sub>2</sub> compd., m. 163-4° (from EtOH) (visible and ultraviolet spectra are given). A lactone (V) obtained from I by catalytic hydrogenation, was reduced with LiAlH<sub>4</sub> to give a diol C<sub>14</sub>H<sub>18</sub>O<sub>2</sub> (VI), b.p. 165-7°, the dehydration of which by refluxing 5 min. with  $\beta$ -Me-C<sub>6</sub>H<sub>5</sub>SO<sub>3</sub>H in xylene yielded an ether (VII) C<sub>14</sub>H<sub>16</sub>O, b.p. 147°, d<sub>4</sub> 0.9681, n<sub>D</sub> 1.4951. A

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hydroxy lactone prep'd. from I by hydrogenation gave by Li<sup>+</sup>H<sub>4</sub> reduction a comixii, m. 159-1°, LXXI. Isolation and properties of prochamazulene from Matricaria chamomilla.

*Chamazulene project*

millia, a further compound of the guanandoids group. Zdeněk Čekan, Vlastimil Herout, and František Sorm. *Chem. Listy* 48, 1071-7; *Collection Czechoslov. Chem. Commun.* 19, 798-804 (1954) (in English).—A bicyclic hydroxy acetoxy lactone  $C_11H_{12}O_4$  (I) contg. 2 double bonds, isolated from dried flowers of *M. chamomilla*, is a source of chamazulene. Extg. 4.3 kg. of an ext. which was dissolved in 2 l. petr. ether, extd. with ten 1000-ml. portions of 2% aq.  $KHCO_3$ , and the aq. layers were extd. 4 times with 500 ml.  $Et_2O$ , and the ether ext. evapd. to give 8.4 g. sirupy residue which yielded 2.5 g. I (prochamazulene), m. 155-60° (from  $Me_2CO$ -iso- $Pr_2O$ ). Hydrogenation of 647 mg. I over  $PtO_2$  in  $AcOH$  gave satd. compds.  $C_{11}H_{14}O_4$ , one eluted with petr. ether, m. 123° (from petr. ether), the other eluted with  $C_6H_6$ , m. 115.5° (from petr. ether). Heating 100 mg. I in 10 ml.  $Et_2O$  with 50 ml. 2%  $AcOH$  30 min. at 50°, extg. the emulsion with  $Et_2O$ , treating the ext. with 2%  $Na_2CO_3$ , extg. the soln. with  $Et_2O$ , acidifying the aq. layer with  $AcOH$ , and extg. it with  $Et_2O$  gave 32 mg. dark blue liquid, *guanulenic acid* (II); its ester, prep'd. by  $CH_3N_3$ , dark blue oil (III). Infrared spectra of I and of the two satd. lactones, and ultraviolet spectra of II, III, and chamazulene are given.  
M. Hudlický

SORM, FRANTISEK

Proteins. XXV. Chemical structures of proteins.  
František Sorm (Czech. Akad. věd, Prague). Chem. Listy 48,  
1954, 137-140; ibid. 48, 1378-16.—Preliminary. XXVII:  
Comparative study of the acidic peptide fractions from par-  
tial hydrolyzates of chymotrypsinogen and trypsinogen.  
Natalia Kell and František Sorm. Ibid. 735-46; cf. C.A.  
48, 134507.—On the basis of analysis of the most acidic pep-  
tides from the partial hydrolyzates of various proteins it is  
concluded that the peptide chains of chymotrypsinogen and  
trypsinogen are comparable in the quantity of cysteine and  
cystine residues and that they differ from other proteins  
studied. The compn. of trypsinogen was as follows (g.  
amino acids/100 g. trypsinogen): aspartic acid 13.6, glu-  
tamic 10.7, arginine 1.0, lysine 10.2, histidine 1.4, serine +  
glycine 10.2, alanine 5.8, valine 9.0, proline 5.5-6.8, leu-  
cine + isoleucine 18.2, phenylalanine 4.4, threonine 6.4,  
cysteine + cystine 6.0, methionine 1.2, tyrosine 8.4, trypto-  
phan 3.7. M. Hudlický—

S.R.P., F.

"Proteins. XXVII. Comparative study of acidic peptide fractions from partial hydrolyzates of chymotrypsin and trypsin." Ceskoslovenska, Morfologie, Praha, Vol. 42, No. 5, May 1954, p. 735.

SO: Eastern European Accessions List, Vol. 3, No. 11, Nov. 1954, L.C.

SORM, FRANCÍSEK

CZECH

Plant substances. III. Substances from *Lactarius deliciosus* L. Věra Benešová, Vlastimil Herout, and František Sorm (Czech. akad. věd, Prague). *Chem. Listy* 48, 662-6 (1954); cf. *C.A.* 47, 7735g; 49, 907c.—EtOH extn. of 23.5 kg. *Lactarius deliciosus* L. gave 1.8 g. mannitol, m. 168°. From the C<sub>2</sub>H<sub>6</sub> ext. (6 g.) were isolated: ergosterol stearate, m. 110.5°,  $[\alpha]_D^{25} -53.4^\circ$  (ergosterol, m. 101°,  $[\alpha]_D^{25} -121^\circ$ ; acetate, m. 174°,  $[\alpha]_D^{25} -93^\circ$ ); 0.40 g. octyl stearate, m. 35.8°, b.p. 180-07° (infrared spectra given); 0.19 g. lactarulene; 0.11 g. verdarulene, m. 04.5°; and lactarulene, m. 03.7°.  
M. Hudlický

SORM, FRANTISEK

I Mechanism of antibiotic action. II. The specific effects of  $\alpha$ -chloramphenicol on the development of seedlings. Frantisek Sorm, Marie Zelinková, and Zora Sormová (Cesk. akad. věd., Praha). Chem. Listy 48, 910-14 (1954); cf. C.A. 48, 4044b.—Differences in intensity and direction of the inhibitory effect of  $\alpha$ -chloramphenicol depend on the nature of the seedlings. Most intensive inhibition of growth and biosynthesis of chlorophyll was found in seedlings with protein reserves. Seedlings of carbohydrate nature are affected less strongly. Oily seedlings show almost no inhibition of growth, but a strong inhibition of chlorophyll synthesis. IV. Accumulation of free alanine in seedlings under the influence of  $\alpha$ -chloramphenicol. Marie Zelinková and Frantisek Sorm. Ibid. 1246-9; cf. C.A. 48, 13801d.— $\alpha$ -Chloramphenicol (I) causes accumulation of alanine in seedlings of various types. The accumulation depends, within certain limits, directly on the concen. of the antibiotic in the cultivation medium. At the highest concen. of I (60  $\gamma$ /ml.), the amt. of alanine in seedlings (5 days old) of wheat increases 12 times, in those of rape seed 8 times, of buckwheat 4 times, and of pea 3.8 times. The common presence of alanine and I in the cultivation medium increases the effect of the antibiotic, whereas alanine alone stimulates growth of pea seedlings, especially the root system.

M. Hudlický

CZECH

Steroids. XIII. Determination of the configuration of 3-dimethylamino derivatives of cholesterol. Jindřík Láber, Václav Černý, and František Šorm (Czech. akademie věd, Prague). Chem. Listy 48, 1009-1012, Collection Czechoslov. Chem. Commun., 19, 1249-57 (1964) (in English); cf. C.A. 49, 359a. - Previously prep'd. 3-dimethylaminocholestanone (I) was proved to have 3 $\beta$ -configuration. Cholestanone (6 g.) in 250 ml. 90% EtOH treated at room temp. with 48 g. KCN and 51 ml. AcOH, pouring the mixt. (after 8 hrs.) into 1 l. H<sub>2</sub>O, extg. the sepd. solid cholestanone cyanohydrin with AcOEt-CHCl<sub>3</sub>, washing the ext. with H<sub>2</sub>O, 3% HCl, and H<sub>2</sub>O, and evapg. the soln. in vacuo at 40° yielded 6.7 g. cyanohydrin which was dried azeotropically and dehydrated by refluxing 4 hrs. with 45 ml. C<sub>6</sub>H<sub>5</sub>N and 5 ml. POCl<sub>3</sub>. The crude 3-cyano-2(or 3)cholestene (5.47 g.) m. 129-30°, [α]<sub>D</sub> 77°, obtained by pouring the dehydrated mixt. into H<sub>2</sub>O and by extg. of the mixt. with Et<sub>2</sub>O. Hydrogenation of 5.6 g. unstd. nitrile in dioxane over 5% Pd-CaCO<sub>3</sub> at 10° and 748 mm. gave 3.7 g. 3 $\beta$ -cyanocholestan (II), m. 140.5-51° (from EtOH). Dintz., II (0.96 g.) with a mixt. of 3.2 g. NaOH, 6 ml. H<sub>2</sub>O, and 52 ml. MeO-CH<sub>2</sub>CH<sub>2</sub>OH until the temp. rose to 125°, refluxing the mixt. 7 hrs., pptg. the Na salt with equal amt. of Et<sub>2</sub>O, decompg. the aq. suspension of the salt with 15% HCl, and extg. the free acid (III) with Et<sub>2</sub>O gave 520 mg. crude and 755 mg.

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Organosilicon compounds. III. Comparison of the reactivities of alkoxysilanes toward the Orgueil reagent.  
Čeněk Rathouský, Vladimír Bafanič, and František Šorm  
(Czech. Akad., věd. Praha). Chem. Listy 1965, 59, 1171  
(1965); Collection Czechoslov. Chem. Commun. 20, 72-81  
(1965) (in Russian); cf. C.A. 49, 161d.—The reactivity of alkoxysilanes toward MeMgCl decreases with increasing mol. wt. and with branching: Et<sub>2</sub>O > PrO > BuO > iso-BuO > iso-PrO > sec-BuO > tert-BuO.

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Sorm, František

Mechanism of antibiotic action. III. Synthesis of some enzymes systems by *Escherichia coli* under the influence of p-chloramphenicol. Dezsőr Grünberger and František Sorm (Czech. Akademie věd, Prague). *Chem. Listy* 48, 1647-53 (1954); cf. *C.A.* 48, 13827c.—No inhibition of synthesis of the enzymes involved in transamination between  $\alpha$ -ketoglutaric acid and aspartic acid, valine, or leucine was observed with *E. coli* (I) under the influence of p-chloramphenicol (II). The inhibition of some amino acid decarboxylases observed previously was thus proved to be unrelated to the inhibition of coenzyme synthesis. The antibiotic does not decrease the formation of the  $\gamma$ -amino-butyric acid- $\alpha$ -ketoglutaric acid transaminase in growing I cells. II does not inhibit the adaptive enzymes of I which take part in  $HCO_3^-H$  metabolism. Partial inhibition of the hydrogenlyase of  $HCO_3^-H$  occurs in such II concns. which already suppress the propagation of bacterial cells.

M. Hudlický

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✓ Steroids. XIV. Rearrangements of basic 3,5-cyclocholestanes. Ludvík Labler and František Sorm (Czechoslovakia, Acad. Prague). *Chem. Listy* 48, 1076-91 (1954). Collection *Czechoslov. Chem. Commun.* 20, 188-91 (1955) (in Russian); cf. *C.A.* 49, 9873a. - Stereospecific rearrangements of substituted 6-amino-3,5-cyclocholestanes lead to 3-substituted 5-cholestenes. Heating HCl salt of 6-dimethylamino-3,5-cyclocholestane (I) (0.5 g.) (m. 198°) at 210° in a stream of dry HCl and triturating the cooled product with Et<sub>2</sub>O gave 0.16 g. of recovered product, and by evapn. of the filtrate, 0.3 g. 3 $\beta$ -chloro-5-cholestene, m. 91-3° (from EtOH and from Me<sub>2</sub>CO). Heating 20 g. 3 $\beta$ -tosyloxy-5-cholestene with 120 mL liquid MeNH<sub>2</sub>, 17 hrs. at 100° in an autoclave, extg. the mixt. with K<sub>2</sub>O, washing the ext. with 5% NaOH, evapg. the solvent, and chromatographing the residual oil (14 g.) yielded 1.6 g. 6-

SORM, FRANTISEK

Effect of anions on brain glutamic acid decarboxylase.  
František Sorm and Tomáš Turský (Czech. akad. věd.,  
Prague). Česk. Listy 48, 1403-8 (1954).—Brain glutamic  
acid decarboxylase is inhibited with anions in the sequence:  
 $\text{AcO}^- < \text{Cl}^- < \text{PO}_4^{3-} < \text{citrate} < \text{Br}^- < \text{NO}_2^- < \text{SO}_4^{2-} < \text{I}^-$ . The  
anion inhibition is of competitive type. The inhibitive ef-  
fect is influenced by pyridoxal phosphate. Application of  
bromides *in vivo* does not change the activity of the decar-  
boxylase *in vitro*. M. Hudlický

CZECH

LXIII. Total synthesis of chamazulene. A simple general synthesis of 1,4,7-substituted azulenes. Ivan Novák, František Šorm, and JIří Šicher (Czech. Akad. ved, Prague), Czech. Čas. Chem. 48, 1648-55 (1954); Collection Czechoslov. Chem. Commun. 19, 1264-73 (1954) (in English); c. C.A. 49, 9084.—A general synthesis of 1,4,7-trialkyl-azulenes has been worked out based on the hydrogenation of a suitably substituted ar-dihydroxybenzosuberene to the said diol, oxidation of the diol to the corresponding dicarboxylic acid, and cyclization to the desired azulene skeleton. Chamazulene was synthesized in this way. 2,3-(MeO)<sub>2</sub>CH<sub>2</sub>COCl and Pr<sub>2</sub>Cd gave 72% 2,3-(MeO)<sub>2</sub>CH<sub>2</sub>COPr (I), b.p. 104°; semicarbazone, m. 184.5° (from MeOH). Refluxing 120 g. I, 61 g. Me<sub>2</sub>NH<sub>2</sub>HCl, 35 g. (CH<sub>2</sub>O)<sub>2</sub>, and 2.5 ml. concd. HCl in 450 ml. EtOH 4 hrs. with efficient stirring, adding 10 g. (CH<sub>2</sub>O)<sub>2</sub>, refluxing 8 more hrs., distg. off most of the EtOH, stirring the cryst. residue several times with Et<sub>2</sub>O, dissolving the crystals in H<sub>2</sub>O, liberating the bases with 400 ml. 10% NaOH, extg. the mixt. with Et<sub>2</sub>O, concd. the ext. in vacuo to 80 ml., and treating the concentrate with dry HCl gave 68 g. 2,3-(MeO)<sub>2</sub>CH<sub>2</sub>CO-CH(CH<sub>2</sub>NMe<sub>2</sub>)<sub>2</sub>HCl (II), m. 141° (from EtOH), and 65 g. recovered I. Treating 21.5 g. II in 60 ml. H<sub>2</sub>O with 40 ml. 10% NaOH, extg. the liberated base with Et<sub>2</sub>O, adding with cooling 20 g. MeI, evapg. the excess MeI and Et<sub>2</sub>O *in vacuo*, stirring the crystals with 100 ml. EtOH, treating the mixt. with NaCH(CO<sub>2</sub>Bu)<sub>2</sub> (prep'd. from 2.2 g. Na and 15 g. CH<sub>2</sub>(CO<sub>2</sub>Et)<sub>2</sub> in 100 ml. EtOH), refluxing the mixt. until no more Me<sub>2</sub>N escaped (8 hrs.), distg. off the EtOH, dissolving the NaI in H<sub>2</sub>O, refluxing the crude ester 2,3-(MeO)<sub>2</sub>CH<sub>2</sub>COCH(CH<sub>2</sub>NMe<sub>2</sub>)<sub>2</sub> with 10 ml. 10% NaOH 2 hrs., adding the mixt., decarboxylating the free acid by heating 20 min. at 170°, and distg. the crude acid *in vacuo* gave 13.6 g. 2,3-(MeO)<sub>2</sub>CH<sub>2</sub>COCH(CH<sub>2</sub>NMe<sub>2</sub>)<sub>2</sub> (III), b.p. 190°. Hydrogenation of 23.7 g. III in 200 ml. AcOH over 3 g. 5% PtLa at 40-90° gave 10.6 g. 2,3-(MeO)<sub>2</sub>CH<sub>2</sub>CH(CH<sub>2</sub>NMe<sub>2</sub>)<sub>2</sub> (IV), b.p. 181°, yielding 23% IV by heating 1 hr. at 30° with 75% poly-

## CZECH

SORM, F.

*Proteins.* XXVIII. Comparison of the arginine peptides from partial hydrolyzates of chymotrypsinogen and trypsinogen. Jiri Vandek, Bořivoj Keil, and František Sorm (Česk. akad. věd, Prague, Czech.). *Chem. Listy*, 48, 1837 (1954); cf. C.A. 48, 13747f.—Chymotrypsinogen (I), trypsinogen (II), and insulin (III) (in 200 cc. soln.) were partially hydrolyzed by heating 6 days at 37° with 10 ml. concd HCl. The arginin peptides were sepd. by passing the hydrolyzates over a column filled with Amberlite IRA-400, and further investigated by means of total hydrolysis and the dinitrophenyl method. Both I and II contain the grouping seryl-arginyl and valyl-arginyl; I differs from II by contg. a third mol. of arginine bound to threonine and alanine. In III the main grouping of glycine and arginine corresponds to the Sanger formula but the grouping of arginine with leucine is in contradiction.

XXIX. Oxidation of pancreatic proteases. Bořivoj Keil. *Ibid.* 1837-41.—Oxidation of I, II, and  $\alpha$ - and  $\gamma$ -chymotrypsins with performic acid is accompanied by denaturation of the proteins and by splitting off small amts. of low-mol. peptides. Hydrolytic fission of peptide bonds during the reaction cannot be prevented. Biol. activity decreases rapidly with oxidation. Two main chains of chymotrypsin seem to be linked not only by disulfide bridges, but by stronger, probably peptide bonds. The oxidation was carried out by dissolving the protein (1 g.) in a mixt. of 40 ml. 90%  $\text{HCO}_2\text{H}$  and 4 ml. 26%  $\text{H}_2\text{O}_2$ , holding at room temp. 20 min., adding 40 ml.  $\text{H}_2\text{O}_2$ , and evapg. to dryness below 50° *in vacuo*. The residue was stirred with  $\text{Me}_2\text{CO}$  (60 ml.), the gummy mass centrifuged, washed twice with 60 ml.  $\text{Me}_2\text{CO}$ , stirred with 60 ml. 0.1N  $\text{NH}_3\text{OH}$ , after 2 hrs. the pH adjusted to 8 with 5N  $\text{H}_2\text{SO}_4$ , the ppt. 0.8 g. centrifuged, the filtrate acidified with 5N  $\text{AcOH}$  to pH 4, dried.

F. Vanecek

from frozen state, the residue dissolved in 8 ml. H<sub>2</sub>O, treated with 8 ml. 50% AcONH<sub>4</sub>, the ppt. of 20 mg. centrifuged, the soln. evapd., and AcONH<sub>4</sub> evapd. at 90° and 0.1 mm., leaving 80 mg. product (A), sol. in H<sub>2</sub>O, mol. wt. approx. 3000. Ultraviolet absorption spectra show an absence of aromatic amino acids. Ultraviolet spectra of I, II, and fractions (A) of both proteins are given.

M. Hudlicky

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SORM, F.

Content of adenosinetriphosphoric acid in seedling of  
*Phaseolus vulgaris*. K. Šebesta and F. Sorm (Czech.  
akad. věd, Prague). ČAS. LÉK. 49, 1060 (1954).  
The content of adenosinetriphosphoric acid was followed  
in bean seedlings, and was found to increase 8-9 times during  
the 5 days of germination, being finally 20 mg. %.

M. Hudlický

SORM, F.

Quinolizine alkaloids. V. Dipole moments and configurations of ( $\pm$ )-*lupinine* and ( $\pm$ )-*spilupinine*. Josef Ratuský, Arnold Reiser, and František Sorm (Czech. Akad. věd, Praha). Chem. Listy 49, 1901 (1955); cf. C.A. 49, 3288. From dipole moment measurements (in D<sub>6</sub>) of ( $\pm$ )-*lupinine* m. 59° (3.07 D.), ( $\pm$ )-*spilupinine*, m. 81° (2.30), ( $\pm$ )-*J.-lupinine* (Ia), m. 50° (2.87) and ( $\pm$ )-*J.-spilupinine* (Ib), m. 30° (2.42), it was deduced that Ia possesses configuration  $\alpha$  and Ib configuration  $\beta$ . A new synthesis of Ia and Ib is described. Hydrogenation of 94.3 g. di-Et [2-(2-pyridyl)ethyl]malonate in 10 ml. AcOH and 30 ml. EtOH over 2 g. PtO<sub>2</sub> 20 hrs. at normal conditions, evapn. of the solvents, washing of the residue with H<sub>2</sub>O and NaHCO<sub>3</sub>, and extn. with Et<sub>2</sub>O gave di-Et [2-(2-pyridyl)ethyl]malonate (II), viscous oil, cyclized to Et octahydro-4-oxo-3-quinolinesuccinate by distn. Treating 8.9 g. II in 60 ml. H<sub>2</sub>O alkalized to pH 8-10 with Na<sub>2</sub>CO<sub>3</sub> with an equiv. amt. of 40% HCHO (2.56 ml.) 3 days at room temp., evapg. the soln. *in vacuo* below 60°, extg. the residue with ether, and evapg. the solvent gave a viscous oil, probably a HOCH<sub>2</sub> deriv. of II. Distn. at 180-200° *in vacuo* gave 3.4 g. (73.6%) Et octahydro-3-quinolinesuccinate (III), b.p. 6-14, 125-35°, b.p. 122-3°. Also obtained in 87.5% (3.0 g.) yield by treating 4.28 g. II with 1.44 ml. 40% CH<sub>2</sub>O in 9 ml. C<sub>2</sub>H<sub>5</sub>N. Shaking 3.8 g. III in 50 ml. N NaOH at room temp. overnight, neutralizing the soln. with HCl, heating it 3 hrs. on the steam bath, alkalining, extg. with Et<sub>2</sub>O to remove octahydro-4-oxoquinolaine; acidifying with HCl to Congo red, evapg. *in vacuo*, dissolving the residue in 50 ml. sats. Et<sub>2</sub>O, satg. the soln. with HCl, repeating the esterification twice more, evapg. the mkt. to dryness, neutralizing the residue with the min. atm. of aq. NaHCO<sub>3</sub>, adding dry Na<sub>2</sub>SO<sub>4</sub>, boiling the ppt. several times with Et<sub>2</sub>O, evapg. the solvent, and distg. the residue gave 2.6 g. (56%) Et octahydro-3-quinolinesuccinate (IV), b.p. 112-14°.

Dissolving 2.8 g. IV in 60 ml. Et<sub>2</sub>O, adding 0.8 LiAlH<sub>4</sub> in 30 ml. Et<sub>2</sub>O, decomp., the mixt. with H<sub>2</sub>O, extg. with Et<sub>2</sub>O and distg. the ext. gave 1.9 g. (85%) 3-(hydraxymethyl)octahydroquinoxaline (mixt. of Ia and Ib), b.p. 05-7°. In the chromatography over 100 g. alk. Al<sub>2</sub>O<sub>3</sub>, the petr. ether fractions gave Ia, m. 59°, and the ether fractions yielded Ib, m. 30°. The dielec. const. were measured at 25°. Also in *Collection Cechoslov. Chem. Commun.* 20, 798-803 (1955) (in English). M. Hudlický

SOFM, F.

Terpenes. LXV. Constitution of lactavaroviolin. Synthesis of 1-ethyl-4-ethyl-7-isopropylazulene and 1-methyl-4-ethyl-7-isopropylazulene. p. 1823

Vol. 48, no. 12, Dec. 1954

CHEMICKE LISTY

Praha, Czechoslovakia

So: Eastern European Accession Vol. 5, no. 4, 1956

SORM, F.

## C Z E C H

Variations in the nucleic acid content in seedlings of *Pisum sativum* in early stages of development under the influence of chloramphenicol. Zora Šormová and František Sorm (Česk. akad. věd, Praha). *Chem. Listy* 48, 1022 (1954).  
—The syntheses of ribonucleic (I) and deoxyribonucleic (II) acids in the first 15 days of development of pea seedlings depends strongly on light and nourishment of the plants. The changes in content of I are relatively small, the content of II increases considerably. Chloramphenicol does not inhibit the synthesis of the nucleic acids to any considerable extent in the pea seedlings, but decreases the rate of development.  
M. Hudlický

SORM, FRANTISEK

CZECHI

✓ Virus. IV. Purification of Rous sarcoma virus by differential centrifugation. Libor Šlechta, Alexander Jakubovíč, and František Sorm (Czech. Acad. Sci., Prague). *Chem. Listy* 48, 1846-9 (1954); cf. *Ibid.* 800.—Differential centrifugation of ext. from Rous hen sarcoma yielded a fraction contg. 0.27% of the total N content of the ext. and was identified as the microsome fraction by means of electron microscopy. L.D.<sub>50</sub> of the fraction based on the N content was 260 times as high as L.D.<sub>50</sub> of the starting ext. V. Effect of adenosine, adenylic acid, adenosinetriphosphate, and guanylic acid on the growth of Rous sarcoma. A. Jakubovíč, L. Šlechta, and F. Sorm. *Ibid.* 1873-7.—Adenosine, adenosinetriphosphate, adenylic acid, and guanylic acid, especially the last two compds., are stimulants for the growth of Rous sarcoma.

M. Hudlický

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SORM, FRANTIŠEK

*✓*  $\alpha$ -Chloramphenicol and its analogs. František Šorm and JHI Gut. Czech. 84,414, Dec. 1, 1958. A simplified method is described starting from the HCl salts of 2-aminoacetophenones which are acylated directly to the *N*-acyl compds. Hydroxymethylation with HCHO in sq. alk. medium yields 2-acylamino-3-hydroxypropiophenones which are reduced by the Meerwein-Ponndorf method to 1-phenyl-2-acylamino-1,3-propanediols. This procedure is shorter by 2 steps than the widely used method (cf. *Mfg. Chemist* 21, 236(1950)) and the final reduction yields only the desired *threo*-diastereoisomer, thus raising the yield by more than 100%. The method can be applied to derivs. arbitrarily substituted in the ring and acylated in the amino group, thus yielding various analogs of chloramphenicol (I).  $\rho$ -O<sub>2</sub>N-C<sub>6</sub>H<sub>4</sub>COCH<sub>2</sub>NH<sub>2</sub>·HCl (1 mole) heated in dry C<sub>6</sub>H<sub>6</sub> with 1.1 moles C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>COCl until it dissolved (6-8 hrs.) yielded 90-95%  $\rho$ -O<sub>2</sub>N-C<sub>6</sub>H<sub>4</sub>COCH<sub>2</sub>NHCOCH<sub>2</sub>Cl<sub>2</sub> (II), m. 144°. II (50 g.) in 250 ml. EtOH treated with 50 ml. 40% aq. HCHO soln. and 0.5 g. NaHCO<sub>3</sub>, and the mixt. stirred until all ingredients dissolved (1-2 hrs.) and dild. with 1000 ml. water gave 68% cryst.  $\rho$ -O<sub>2</sub>N-C<sub>6</sub>H<sub>4</sub>COCH(NHCOCH<sub>2</sub>)<sub>2</sub>CH<sub>2</sub>OH (III), m. 121°. Al(OCHMe<sub>2</sub>)<sub>3</sub> (20 g.) in 200 ml. boiling dry iso-PrOH treated gradually with 20 g. III in iso-PrOH, the iso-PrOH and acetone distd. off, the residue boiled briefly with water, and the crude I extd. with Et<sub>2</sub>O and crystd. yielded 50% product m. 150-1°.

L. J. Urbánek

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*Chem.*

~~SECRET SOURCE DATA~~  
DR M. FRANTISEK

Azulenes from *Artemisia* plants. Vlastimil Herout and  
Prantilek form. Czech. 85,240, Dec. 1, 1955. A mixt. of  
3 kg. dry finely cut *Artemisia* and 1 kg. NaOH was dissolved  
in 20 l. water, heated for 18 hrs. on a steam bath, and  
slowly acidified with a soln. of 1.35 kg. 94% H<sub>2</sub>SO<sub>4</sub> in 2 l.  
water. The mixt. was steam-distd. and the azulene layer  
sepd. to give 17.3 g. of a crude mixt. contg. 31% azulenes  
(I). Sepn. was facilitated by addn. of 1 l. of ligroine which  
was then distd. off *in situ*. The residue was dissolved in  
100 ml. of ligroine and extd. with 30 ml. of concd. HCl.  
The acid soln. was dild. with 800 ml. of icewater and shaken  
with 250 ml. of Et<sub>2</sub>O. After evapg. the Et<sub>2</sub>O, the residue  
(5.8 g.) was taken up in ligroine and chromatographed over  
00 g. Al<sub>2</sub>O<sub>3</sub>, yielding 5.3 g. of a pure mixt. of I, bp 150-80°.

L. J. Urbánek

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*Similarities in the structure of proteins. B. Keil and P. Sorm (Czech. Acad. Sci., Prague). Congr. intern. biochim., Résumés communs., 3<sup>e</sup> Congr., Bruxelles 1955, 16-17 (1955) (in German); cf. C.A. 48, 13747f.—Proteins of similar origin and function should also show structural similarities. Chymotrypsinogen (I) and trypsinogen (II) on partial hydrolysis both yielded peptides contg. seryl-arginyl and valyl-arginyl groups. In I, the arginine is bonded to alanine and threonine. The similarities of I and II and disialoharmonies in relation to other proteins were demonstrated. In studies of serum albumins, hemoglobins, and myoglobin, the proteins tended to be similar in structure in phylogenetically related species of animals (no details). Chem. similarities were related to antigenic similarities.*

*W. C. Tobie*

*✓* The nature of the endogenous substrate in Ehrlich ascites tumor. L. Šlechta, A. Jakubovič, and F. Sorm (Czech. Acad. Sci., Prague). *Congr. intern. biochim.*, Résumés communs., 3<sup>e</sup> Congr., Brussels 1955, 126 (in English); cf. C.A. 49, 5645d.—Another paper (cf. C.A. 49, 10493d) is supplemented. The respiratory quotient (RQ) for endogenous respiration was detd. in Ehrlich ascites tumor cells with and without glucose and with and without iodoacetic acid or DL-glyceraldehyde (inhibitors of carbohydrate metabolism). In all cases the respiration and RQ were nearly identical. Apparently the endogenous substrate is lipide. Changes in the fat content, and the fact that the cells contain no glycogen furnish further proof that lipides not carbohydrates are the substrate. W. C. Tobie

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SUR R<sup>2</sup>, F.

✓ A further chamazulene precursor: the bitter principle of *Artemisia absinthium*. F. Sorm, L. Norotthy, and V. Herout (Czechoslovak Acad. Sci., Prague). *Chemistry & Industry* 1955, 669; cf. *C.A.* 49, 8241c.—Extrn. of *Artemisia absinthium* with petr. ether or EtOH yields a compd. (I), m.p. 152-8° (decompn.) (from a.c.). Alk. hydrolysis of I in the presence of air, followed by acid distn., yields 13% chamazulene. The infrared spectra of I and anabainetine (II) ( $C_{14}H_{16}O_3$ ), m.p. 260°, are almost identical, but their relation is not clear. II is formed from I during extn. The infrared spectrum of II exhibits the characteristic infrared absorption of a  $\gamma$ -lactone.

J. A. Gilea

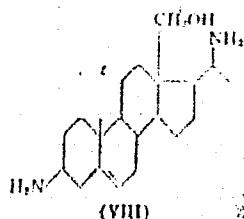
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SvRM, F

*Structure of halotetraline.* L. Čeláček, V. Černý, and E. Šimíček (Czech. Acad. Sci., Prague, University of Tübingen)

**Dissertation:** The author is of the opinion that halotetraline (III) presented a pregnane nucleus (C. I. 48, C47) has now been verified. Dihydrotetrahydrohalotetraline (II) and  $\text{SO}_2\text{Cl}$  gave a *quaternary chloride hydrochloride* which gave the corresponding "disporate" (III), m. 218-9° (from  $\text{Et}_2\text{O}-\text{H}_2\text{O}$ ); II with the *h. trichloro*, and the *quaternary base* was converted to the *anilide* (IV). IV was then easily decarboxylated to give dihydrocortisone, m. 137°, [ $\alpha$ ]<sub>D</sub> 58.4° ( $\text{CHCl}_3$ ), and dihydrocortisone amine, m. 141.5°-141.7° ( $\text{CHCl}_3$ ). II and *MeCO-COOH* gave a *quaternary p-toluenesulfonate* (V), m. 208-211° (from  $\text{MeOH}-\text{Me}_2\text{CO}$ ), [ $\alpha$ ]<sub>D</sub> 23° ( $\text{MeOH}$ ); V with  $\text{NaI}$  in  $\text{Me}_2\text{CO}$  gave  $\text{C}_21\text{H}_{21}\text{N}_3$  (VI), m. 301-3° (from  $\text{MeOH}-\text{Me}_2\text{CO}$ ), [ $\alpha$ ]<sub>D</sub> 31° ( $\text{MeOH}$ ); VI with  $\text{MeI}$  gave dihydrocortisone-23*Me* (VII), m. 319-20° ( $\text{EtOH}-\text{Me}_2\text{CO}$ ), [ $\alpha$ ]<sub>D</sub> 23° ( $\text{MeOH}$ ). Hofmann degradation of VII gave dihydrocortisone amine, m. 122-4° ( $\text{Me}_2\text{CO}-\text{H}_2\text{O}$ ). VII is proposed as the structure for I.



Harry L. Yale

W.M. S.

SHOEM, F. Some achievements in the chemistry of sesquiterpenes. p. 441  
Vol. 3, 1955 INVESTILA. Sofiia, Bulgaria

SOURCE: East European Accessions List (EEAL) Vol. 6, No. 4--April 1957

HOLUB, M.; HEROUT, V.; SORM, F.

Synthesis of  $\alpha$ -bisabolol - a spasmolytically active sesquiterpenic alcohol. Cesk.farm. 4 no.3:129-131 Apr 55.

1. Oddeleti prirozenych latek, Ustav organické chemie, Československa akademie ved, Praha.

(ALCOHOLS,

$\alpha$ -bisabolol, synthesis, spasmolytic eff.)

(MUSCLE RELAXANTS,

spasmolytic eff. of  $\alpha$ -bisabolol alcohol)

SORM

LACKOVA, E., MUDr; HELSKA, M., MUDr; SORM, F., akademik; SVRJCAR, Jiri, MUDr

Effect of testosterone propionate on infantile dystrophy. Cesk. pediat. 10 no.2:107-113 Mar 55.

1. Z I detske klin.; predn. prof. Dr. J. Svejcar.  
(ABNORMALITIES

systemic, causing dystrophy in inf., ther. testosterone propionate)

(TESTOSTERONE, derivatives  
propionate, ther. of dystrophies in inf.)

(KIDNEYS, diseases  
exper. dystrophy in rats, eff. of testosterone propionate)

SOKH / CZEW

10045° - Organosilicium Compounds. Organosiliciumverbindungen. III. Comparison of the Reactivities of the Alkoxysilane. With the Grignard Reagent. Vergleich der Reaktivitäten der Alkoxysilane mit dem Grignardreagenz. (German)

J. Barthovský, V. Bláha and F. Šorm. Collection of Czechoslovak Chemical Communications, Vol. 27, no. 1, Feb. 1955, p.

72-81.

Reactivity of various epoxy-groups bound to the Si<sup>4+</sup> during a reaction with methyl magnesium chloride. Intermediate products described. Graphs, tables. 20 ref.

At open

SORM, F.; AND OTHERS

On terpenes. LXV. The constitution of lactaroviolin; synthesis of 1-ethyl-4-methyl-7-isopropylazulene and 4-ethyl-1-methyl-7-isopropylazulene. In English.  
p. 227

Vol. 20, no. 1, Feb. 1955  
SBORNÍK ČESkosLOVATSKIHH KHLIMICHESKIKH RABOT  
Praha, Czechoslovakia

So: Eastern European Accession Vol. 5, No. 4, April 1956

SORM, F.; TURSKY, T.

Effect of anions on the glutamic acid decarboxylase of brain tissue. In Russian.  
p. 290.

Vol. 20, no. 2, Apr. 1955  
SBORNIK CHEKHOVATEKIKH KHMICHESKIKH RABOT  
Praha, Czechoslovakia

So: Eastern European Accession Vol. 5, No. 4, April 1956

~~HANSON~~  
SORM, F.

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Amino acids and peptides. XV. Preparative isolation  
of some amino acids from gelatin hydrolysate. Václav  
Holečkovský and František Šorm (Čech. akad. věd  
Prague, Chem. Listy 1965, 59, 243-5; Collection Czechoslov. CH  
Chem. Commun. 20, 686-62 (1965) (in Russian); cf. C.A.  
49, 11040d.—L-Proline (I), D,L-hydroxyproline (II), L-  
valine (III), and a mixt. of optically active leucine and iso-  
leucine (IV) were prepd. from gelatin by hydrolysis, ester-  
ification with iso-PrOH, distn. of the esters, hydrolysis of  
the esters, and partition chromatography of the acids on  
cellulose. Gelatin (250 g.) was dissolved in 750 ml. concd.  
HCl, the soln. refluxed 10 hrs. at 130-40°, decolorized with  
activated C, and evapd. to a syrup. The residue (331 g.)  
was dissolved in 750 ml. iso-PrOH at 70°, the soln. satd.  
with HCl at 0°, refluxed 5 hrs., and the alcohol distd. off  
at 20°. The esterification was repeated twice. The resid-  
ual mixt. of ester hydrochlorides was treated at -7° with  
an equiv. amt. of iso-PrONa, then with 2 l. Et<sub>2</sub>O, and the  
soln. distd. over a column at reduced pressure and 40° to  
give 160.3 g. of crude esters (out of a 162-g. portion of gel-  
atin). The esters were distd. over a 7-plate column and  
sepd. into 5 fractions. A fraction (13.6 g.) was refluxed 8  
hrs. with 272 ml. 0N HCl, the hydrolysate evapd. (13.1 g.),  
and the residue dissolved in H<sub>2</sub>O and passed over an ion  
exchanger. The eluate was evapd. to dryness *in vacuo*  
and the residue chromatographed on cellulose, using BuOH-  
H<sub>2</sub>O-AcOH (4:6:1) for the isolation of I, III, and IV, and  
PhOH satd. with H<sub>2</sub>O for the isolation of II. Out of 100 g.  
gelatin, there were obtained 0.85 g. I, 2.1 g. II, 30.2 g. III,  
and 2.1 g. IV.  
M. Hudlický

(1)

SORM, FRANTISEK

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~~Quinolizine alkaloids. V. Dipole moments and configurations of ( $\pm$ )-3-lupinine and ( $\pm$ )-3-epilupinine.~~ Josef Ratajsky, Arnost Reiser, and Frantisek Sorm. Collection 77  
Czechoslov. Chem. Commun. 20, 788-803 (1955) (in English).  
See C.A. 49, 14780i. E. J. C.

(2)

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